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THE ELEMENTARY PRINCIPLES OF CHEMISTRY

BY

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PART II

EXPERIMENTAL ILLUSTRATIONS

"I seek to know and to enjoy at once"
ROBERT BROWNING in *Paracelsus*

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1901

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RECOMMENDATIONS AS TO NOTES

THE making of notes is a very important item in the study of a subject in the laboratory. The student should early in his course form the habit of writing them carefully, thoughtfully, and systematically. The aim should be to describe the *essential* features of method and of apparatus, then the things observed, then the things learned from the experiment; for every experiment is designed to teach something. The aim should also be to make the description clear, exact, and simple. The following details of plan are recommended: To use a book about six by nine inches in size, one whose leaves will lie flat when opened, with plain, unruled pages; to enter notes only on the right-hand page, leaving the left-hand for topics, corrections, additions, and the numerical work of calculations; to enter *always* the particular topic which the experiment is to illustrate; as a minor item, to enter the date of work; for convenience, to have the name of the owner on the outside of the cover. The notes should be written in final form in the laboratory, and not copied.

It is particularly urged that the student should read *through* the directions for an experiment before starting upon its performance, and that the experiment should precede the consideration of the corresponding topic in Part I.

THE ELEMENTARY PRINCIPLES OF CHEMISTRY

PART II

EXPERIMENTAL ILLUSTRATIONS

CHAPTER I

INTRODUCTION

Read Part I, Nos. 1–6, before beginning the experiments.

1. Physical Properties of Sulphur

Observe as to odor, color, hardness, form (crystalline —that is, regular geometric form). 1–6*

To determine its specific gravity: Weigh carefully to tenths about 10 grams of dry sulphur, in small lumps, free of dust. Weigh a test-tube, filled with water and corked. Put the sulphur in the test-tube, refill with water, cork, and weigh (see Appendix, 1). 7

The specific gravity of sulphur equals the weight of sulphur divided by the weight of an equal volume of water. Calculate the specific gravity from the observations made.

Suggest another method for solids; also one suitable for solids which are soluble in water; a method for liquids; for gases. 8

Electrification.—Rub a large lump briskly on the dry towel, or, better, on some woolen stuff. It becomes electrified and capable of attracting particles, like bits of paper. 9

* The marginal numbers are the same for the same topics in both Part I and Part II.

10 **Effects of heating.**—Heat in a dry test-tube some lumps of *dry* sulphur, the test-tube being about half filled. It is well to make a holder by folding a strip of paper once or twice upon itself, to wrap this about the test-tube and to seize the ends close to the tube with the fingers, or, better, with the tongs (see Appendix, 2). Apply the heat gradually by passing the tube slowly back and forth through the flame. Have a vessel filled with water on the table close at hand. Should the tube crack and the sulphur take fire, hold quickly over the water. Let the temperature rise slowly while the sulphur is *melting*. Note the color. Keep the surface of the liquid in gentle motion; note the *partial*
11-12 *solidification*. Continuing the heat, note the *second liquefaction*, the beginning of *boiling*, and the white dustlike deposit (*sublimate*). Now, as the sulphur is beginning to boil, move the burner to one side; carefully and slowly turn the melted sulphur into the water, moving the tube to and fro so as to string it out. Set aside the tube with the small residue of sulphur and observe it from time to time as it cools (*solidification* and *crystallization*). Examine the portion which has been suddenly cooled in the water (*allotropism*), comparing it with the original. Select a good sample of it, and, without lumping it together, dry it by pressing and rubbing first between folds of the towel, then between absorbent papers (filter papers). Weigh the sample carefully and leave it for twenty-four hours. Does it change in weight in passing from the *plastic amorphous* (see Part I, No. $21/_6$) to the *brittle crystalline* form?

12/1 *Crystallization from fusion* (see No. $21/_4$, Part I).—Fuse some dry sulphur in a crucible, keeping the temperature as low as will suffice to melt; let it cool slowly until a crust has formed; puncture the crust and pour out the remaining liquid; break the crucible and note the appearance of the crystals known as *monoclinic* sulphur; examine them twenty-four hours later. (It is recommended that the instructor do this experiment for the class.)

12/2 *Crystallization from solution* (see No. $21/_4$, Part I).—Dissolve some sulphur in carbon disulphide to saturation, or nearly so, decant (that is,

drain off) or filter the liquid and allow it to evaporate in the hood. Examine the crystals known as *orthorhombic* sulphur; compare with those of the preceding experiment (*dimorphism*). Do they change on standing? As carbon disulphide is very volatile and combustible, great care should be taken to have *no flame near.* (It is recommended that the instructor do this experiment for the class.)

2. Chemical Properties of Sulphur

Burning or combustion.—Ignite a small lump of sulphur (half the size of a pea) on a suitable surface (an earthen saucer serves well). Describe the phenomenon. The gaseous product is *sulphur dioxide;* observe its odor *cautiously,* and its effect on litmus—conveniently done by sticking a piece of wet, blue litmus paper to the bottom of a beaker and inverting the latter over the burning sulphur. Compare the sulphur dioxide in these respects with the sulphur. If you have a sample of the plastic sulphur, burn and test this in the same manner. 13

EXPLANATORY NOTE.—An invisible gas, namely, *oxygen,* a constituent of the atmosphere, takes part in this change, combining with the sulphur and forming the sulphur dioxide, which is quite different from both the sulphur and the oxygen.

Behavior with zinc.—Weigh out carefully 3.20 grams of finely divided sulphur (sulphur sublimate, commonly called sulphur flowers); also 6.50 grams of finely divided zinc (zinc dust). Mix these thoroughly in a mortar, so that the original powders can not be distinguished (see Appendix, 5). Take out a small portion, about as much as could be heaped on a five-cent piece (putting the remainder well to one side); heat this strongly on the earthen saucer or on the asbestos board by turning the gas flame down upon it, or, still better, on the spatula blade by thrusting it into the flame. *Do this cautiously, as it may flash up somewhat like gunpowder. In experiments like this, the face should never be held over the material.* Describe the phenomenon, note the ashlike product, compare it with the original sub- 13/1

stances, sulphur and zinc, by inspection. Does it melt and burn like sulphur?

(*a*) Put a little of the mixed powders from the mortar in a test-tube, half fill with water, then, closing the tube with the thumb, shake it vigorously, and finally let the powder settle (see Appendix, 4). Does the powder dissolve? Do you see the sulphur tend to separate from the mixture with the zinc? In a similar manner shake some of the ashlike product with water. Does it dissolve? Do you see any tendency of the sulphur to separate from the zinc? Does zinc dissolve in water? Does sulphur dissolve in water?

(*b*) Put a small quantity of the ashlike product in a test-tube and add to it a few drops of *hydrochloric acid*, better designated as *hydrogen chloride* (see Appendix, 3). Note the phenomenon of *effervescence* (bubbles of gas in the liquid; how does it differ from boiling?), indicating the formation or liberation of a gas not previously apparent. Note the odor of this gas (*with care, for the substance is very poisonous*) and its effect on a bit of filter paper wet with a solution of lead acetate, and held at the mouth of the test-tube. Does the ashlike product disappear in the acid? If not, warm the tube slightly.

Compare this with the behavior of a little zinc treated in another test-tube with hydrogen chloride in a similar way (does the zinc powder disappear?); also of sulphur thus treated; also of the mixed zinc and sulphur (does this powder disappear?).

EXPLANATORY NOTE.—In this experiment you have brought about a chemical change. Starting with zinc and sulphur, mixing them intimately and applying heat, you have caused the zinc and the sulphur to disappear, and in their place you have a substance which is distinctly different from each of the original substances. This is a change of identity, a chemical change; and with this meaning, the new substance, which for the moment may be designated x, is said to *contain* zinc and sulphur, and the latter are said to be *combined* in the substance x. To the question, Does the substance x contain nothing but zinc and sulphur? the answer will be suggested by the next experiment.

Behavior with iron.—Weigh out carefully 3.20 grams of 13/2
sulphur powder; also 5.60 grams of finely divided iron (iron dust). Mix them thoroughly in the mortar. Take out a portion, ignite it, and compare the product with the iron, with the sulphur, and with the mixture by inspection and by its behavior with hydrogen chloride, as in the preceding experiment. Does the gas which is obtained have the same odor and action on paper wet with lead acetate as in the preceding?

(*a*) Vary this procedure as follows: Put a portion of the mixed iron and sulphur in a dry test-tube, and cork the latter; weigh it and its contents carefully to tenths; then, holding it nearly horizontal, tap it gently so that the powder spreads out in an elongated pile from the bottom of the tube; let the cork lie loosely in the mouth of the test-tube, and heat the other end in the flame just enough to start the action; then remove from the flame. When the action has ceased, let cool, and weigh again with contents. With a little care, you will be able to do this without losing anything from the tube during the operation. Save the product.

Explanatory Note.—Again you have a chemical change. The substance produced (*y*) *contains* iron and sulphur as constituents, but is neither iron nor sulphur; moreover, it contains *nothing but these*, since what you have in the tube after the change weighs no more than the iron and the sulphur which you put in. Iron and sulphur then must be the sole constituents of the substance *y*; zinc and sulphur are likewise the sole constituents of the substance *x*. The first is therefore named *iron sulphide*, and is said to be *composed* of iron and sulphur, and the second is *zinc sulphide*, and is *composed* of zinc and sulphur. The changes by which these are produced fall under a common type, which may receive a general expression, thus: Substance A and substance B became substance A B (*composition*); or, to express it more concisely,

$$A + B = A\,B.$$

But you have further illustration of chemical change in the production of a peculiar gas by the action of the hydrogen chloride on both zinc sulphide and iron sulphide. This gas is clearly very different from all these other substances. You have noted the same odor and the

same action on the lead paper in both cases, and, in fact, the same gas is produced by both substances. If this is so, evidently neither iron nor zinc can enter into its composition. But the sulphur of one or of the other does so enter, yet the gas is markedly different from sulphur itself and from the gas produced by burning sulphur (see Exp. 13). The sole other constituent of this peculiar gas is hydrogen, which comes from the hydrogen chloride, of which also it is a constituent. The gas is therefore named *hydrogen sulphide.* It is also known as *hydrosulphuric acid.*

13/3 **To show that hydrogen sulphide contains sulphur:** Place about 1 gram (not more than 2) of the iron sulphide, made in the previous experiment, in the gas generator (see Appendix, 6–9) with just enough water to seal the thistle-tube. Drop the delivery tube into a test-tube about one third filled with *nitric acid* (handle this substance with care, for it is very corrosive *), so that the gas shall bubble through this liquid. Pour a few drops of hydrogen chloride into the thistle-tube of the generator, so as to produce a quick stream of bubbles through the liquid. Place a bit of paper wet with lead acetate over the mouth of the test-tube. As the gas from the generator passes through the nitric acid you see small particles of white or yellowish substance appearing, which, when the action has ceased, or nearly so, will have collected into a mass from which you may rinse the acid with water, and which you may easily recognize as sulphur by inspection and by burning. At the same time the lead paper may, perhaps, show no blackening, since the sulphur is produced only at the expense of the hydrogen sulphide, although enough of the latter to stain the paper may escape destruction by the nitric acid.

EXPLANATORY NOTE.—Hydrogen sulphide, therefore, contains sulphur, and, whenever this gas is produced, it may be taken as evidence of sulphur as a *constituent* in the substances from which it is produced.

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* If nitric acid is spilled on the skin, clothing, or table, the spot should be as quickly as possible moistened with ammonia (side-table), and then rinsed with water.

QUESTIONS.—How can you differentiate (that is, distinguish by differences) between sulphur, and oxygen, and sulphur dioxide? Between zinc, and sulphur, and the mixture of zinc and sulphur, and zinc sulphide? Between iron, and sulphur, and the mixture of iron and sulphur, and iron sulphide? What constitutes the difference, by definition, between a mixture and a chemical compound? (see Nos. 13 and 28, Part I.) How do you bring about the chemical change between sulphur and air, between zinc and sulphur, between iron and sulphur? What evidence by observation have you that heat is produced by these three changes? Does zinc sulphide, made by heating zinc and sulphur, change back to zinc and sulphur by cooling? Does iron sulphide reverse the change by cooling? Is the change from iron and sulphur mixed to iron sulphide accompanied by change in the total weight? (see No. 14, Part I.)

3. Additional Illustrations of Chemical Change

Composition.—Put in a dry test-tube about 1 gram of lead 15/1
dust and 0.2 of a gram of iodine (iodine must be weighed on glass or paper, as it corrodes metal); cork the tube and weigh it with its contents carefully; let the cork lie loosely in the mouth of the test-tube and warm the mixture very slightly, at first barely touching the flame with the bottom of the tube. When the first action is over, the substance may be heated until it melts. A little of the iodine may escape the change, making a purple vapor and a dark deposit on the upper part of the tube. When the tube is cool, weigh it with its content. Describe the phenomenon, and compare the yellow substance with the lead and with the iodine. Preserve a sample of it for future comparison.

EXPLANATORY NOTE.—From lead and iodine is produced a substance which does not give off a purple vapor, nor impart a brown color to water, as does iodine, and which is clearly neither iodine nor lead; yet by the test of weight it can contain *nothing more* than iodine and lead. This bright-yellow substance is named *lead iodide*. Does it dissolve in water?

Composition.—Put about 0.3 of a gram of magnesium 15/2
ribbon, or powder, in the porcelain crucible (see Appendix, 11). Weigh carefully the crucible with its content

and its lid. Support the same on the pipe-stem triangle, and the latter on the iron ring of the stand. Apply the full heat of the flame to the bottom of the crucible for ten or fifteen minutes. Raise the lid slightly to observe what is taking place, but do not let any of the white smoke escape. During the last few minutes the lid should be entirely removed. Let the crucible cool, and weigh it again with content and lid. How do the two weights compare? Describe the phenomenon and compare the substance after heating with the original.*

EXPLANATORY NOTE.—The bright metallic ribbon or powder is changed, simply by heating in contact with the air, to a very different substance—a grayish chalklike powder; but this weighs *more* than the original. It must be, therefore, that something besides magnesium is contained in the product. This "something" comes from the air, is an invisible gas, and is named *oxygen*. The white powder is named *magnesium oxide*. Try also heating a short piece of the ribbon directly in the flame, holding it with the tongs; likewise a little of the oxide.

15/3 **Composition.**—In a similar manner heat in the crucible about 2 grams of zinc dust, weighing the crucible and contents before and after the ignition. Compare the weights and describe the phenomenon. *Zinc oxide* is thus formed—yellow while hot, white when cold. Try also heating a little of the zinc dust on the spatula blade. Preserve a little of the zinc oxide for future comparison.

15/4 **Composition.**—Spread about 4 grams of lead dust on the crucible lid, weighing the whole; then ignite over the flame for fifteen or twenty minutes and weigh again. The reddish-yellow powder is *lead oxide*. Preserve a little of this.

EXPLANATORY NOTE.—The statement made concerning magnesium oxide applies also to zinc and lead oxides.

These four instances of *composition* are changes of the general form: Substance A and substance B become substance A B; or

$$A + B = A\,B.$$

* Clean the crucible by using a few drops of hydrochloric acid, warming gently, then rinsing with water. Scouring with sand may be helpful.

QUESTION.—Have you evidence that, when magnesium changes to magnesium oxide, and zinc to zinc oxide, heat is produced?

Decomposition.—Take a quantity of lead nitrate, about 1 gram (twice the size of a pea), pulverize finely in a clean, dry mortar, shake the powder into a clean, dry test-tube, wipe off any particles adhering to the upper part of the tube, cork, and weigh the tube and contents. Then, holding the tube nearly horizontal (paper holder), with the cork loosely placed in its mouth, warm the substance gently until the snapping (*decrepitation*) ceases and the powder melts quietly; remove the cork and continue the heating until the bubbling ceases and the glass begins to soften. Note the effect of the brown gas on wet, blue litmus paper. Let cool, and weigh the tube and contents, including the cork. Break the tube and examine the reddish-yellow residue, *lead oxide*. Save a little of this for future reference. 16/1

QUESTIONS.—Does the residue dissolve in water? Does the lead nitrate dissolve in water? Does the brown gas condense at all on the upper part of the test-tube as you saw in the heating of sulphur? (see Exp. 11.) How does the weight of the residue compare with that of the lead nitrate?

EXPLANATORY NOTE.—In this experiment you see one substance yielding at least two others, distinctly differing from each other as well as from the original; and at least one of these—that is, the residue in the tube—weighs *less* than the original, which has disappeared, although none of the latter, unchanged, has passed out of the tube. The gas which has passed out, named *nitrogen tetroxide*, and the residue, named *lead oxide* (see Exp. $15/_4$), which is left in the tube, must have been *contained* in the lead nitrate which has been destroyed; the latter has been broken up into at least two other substances, each of which weighs less than the original. Such a change is called *decomposition*. It may be described under the general type: Substance A B becomes substance A and substance B; or

$$A\,B = A + B.$$

Decomposition.—Place about 1 gram of zinc nitrate in a test-tube, weigh the tube and contents, then heat. Describe the phenomenon, and note the clear liquid which condenses 16/2

on the upper part of the tube. This is water; it is slowly driven from the tube by heat. Later, note the brown gas, which appears as in the preceding experiment. This is nitrogen tetroxide, and it in turn is driven from the tube. Finally, there is left a powder which no longer melts, and which is yellow while hot and white when cold. This is zinc oxide. Weigh again the tube and contents. Examine the residue left in the tube (comparing with the substance in Exp. $15/_3$), and save a portion for future tests.

QUESTIONS.—Does the residue weigh more or less than the zinc nitrate from which it comes? Does it dissolve in water? Does zinc nitrate dissolve in water?

EXPLANATORY NOTE.—You see the substance, zinc nitrate, destroyed by heat, yielding at least three distinctly different substances—water, nitrogen tetroxide, and zinc oxide; and the last of these, and by inference each of the others also, weighs less than the original substance, and all of them must have been contained in the original zinc nitrate. This change falls under the same type as the preceding:

$$A\ B\ C = A + B + C.$$

17/1 **Substitution.**—(*a*) Place a small quantity of granulated zinc, about 0.5 of a gram, in a test-tube; add to it a few drops of hydrochloric acid (hydrogen chloride). Note the effervescence which implies the liberation of a gas. Close the tube with the thumb for a few seconds, then open it, holding its mouth to the gas flame. Note what takes place.

EXPLANATORY NOTE.—The colorless invisible gas liberated in this change burns with an almost invisible flame, and makes with air an explosive mixture. This causes the slight sound when the content of the tube is ignited. Larger quantities may be dangerously explosive, and this fact should always be in mind when dealing with this gas which is named *hydrogen*.

(*b*) Next, place 1 gram of granulated zinc in a small evaporating dish which, with a small glass stirring-rod (see Appendix, 12), has been previously weighed (to tenths is sufficient). Add about one quarter of a test-tubeful of hydrochloric acid, in portions at a time, warming some-

what the contents of the dish. Holding a lighted match at the surface of the liquid while the bubbles of hydrogen are breaking, will again show the combustibility of this gas. When the zinc has disappeared except a few black specks, and the bubbles of gas no longer appear, increase the heat somewhat, holding the burner in the hand and touching the tip of the small flame to the bottom of the dish from time to time, as may be needed to keep the liquid quietly boiling (see Appendix, 12). This soon thickens and deposits at the edges. Continue the heating, avoid spattering as much as possible, and soon white fumes appear, and the substance solidifies if cooled. At this point cease the heating and, when the dish is cool, weigh it with its contents. Describe the substance in the dish. Does it weigh more or less than the zinc taken? Let some of it remain exposed to the atmosphere for a short time and note the change.

EXPLANATORY NOTE.—Hydrochloric acid contains as sole constituents hydrogen and chlorine, the material which you use being this substance dissolved in water. When this is brought in contact with zinc a change takes place, and, when this is complete and the liquid boiled to dryness, the product just seen is the result. Now this weighs more than the zinc, nearly double, and, therefore, must contain something besides zinc; yet it can not contain anything more than zinc, hydrochloric acid, and water; indeed, it can not contain all of these, for hydrogen has passed into the air, as you have seen, and the water also has been boiled away. In fact, the residue in the dish is a substance which contains as sole constituents the zinc used and the chlorine previously contained in the acid, although it is very different from each of these. It is named *zinc chloride.* Exposure of this to the air for a short time will show you incidentally one of its properties; it absorbs water from the atmosphere and liquefies in consequence (*deliquescence*). In this change it is seen that a constituent of hydrochloric acid—namely, hydrogen—leaves the substance, and zinc may be said to take its place; so that, starting with zinc and hydrogen chloride, you obtain hydrogen and zinc chloride. Such a change is called *substitution.* It may be represented by the general form:

$$A + BC = B + AC.$$

17/2 **Substitution.**—*Optional experiment.*—(*a*) Place about 0.5 of a gram of granulated zinc in a test-tube, add dilute sulphuric acid, warm, and test the gas at the flame as in the preceding experiment.

(*b*) Next place about 3 grams of granulated zinc in the evaporating dish, add about one test-tubeful of dilute sulphuric acid (one volume of concentrated acid to four of water) and an equal volume of water, and warm. When the zinc has disappeared, or nearly so, filter (see Appendix, 13), boil down the liquid somewhat, and set aside to crystallize. Crystals of *zinc sulphate* are thus obtained. The relation of this substance to sulphuric acid is similar to that of zinc chloride to hydrochloric acid.*

17/3 **Substitution.**—Place about 3 grams of lead powder in a small evaporating dish, add about one quarter test-tubeful of nitric acid and an equal volume of water. Heat gently until the action ceases, filter the liquid, and set it aside to crystallize. When there is a considerable deposit of crystals, drain off the liquid, and throw the crystals on two or three thicknesses of filter paper to absorb the adhering liquid. These are crystals of *lead nitrate.* Heat a portion of these and compare the result with that in Nos. $15/_4$, and $16/_1$. Save a portion of the crystals, labeled, for later use.

17/4 **Substitution.**—*Optional experiment.*—Dissolve about 10 grams of crystallized copper sulphate in about 50 cubic centimeters (see Appendix, 14) of boiling water, using the larger evaporating dish. Add to this 4 grams of granulated zinc, and boil the liquid gently until the blue color has entirely disappeared. Filter; concentrate the liquid to about one third its volume; filter again if necessary, and set aside to crystallize. What is the reddish-brown powder

* The irritation which may be caused by the fumes and spray in this experiment, if many are working it at the same time, may be relieved by inhaling moderately the fumes from the bottle of ammonium hydroxide (side-table).

which appears when the zinc is added? Add a few drops of nitric acid to this powder after filtration. What is the substance which crystallizes in the filtrate?

EXPLANATORY NOTE.—Copper sulphate bears the same relation to copper and sulphuric acid that zinc sulphate bears to zinc and sulphuric acid (see Exp. $17/_2$)—that is, copper has taken the place of hydrogen as a constituent in sulphuric acid. When zinc is brought in contact with the solution of copper sulphate, the copper, in turn, is displaced, appearing as a powder, which, with nitric acid, reproduces a blue solution; while the zinc, with the sulphuric acid, forms zinc sulphate, crystallizing as seen in Exp. $17/_2$. Iron may likewise thus substitute itself for copper as constituent. If the action is allowed to take place slowly in dilute solution, the copper may be deposited as a film on an object of iron, such as a knife-blade. This is called "plating."

Double exchange.—The purpose of this experiment is to 18
illustrate a reaction of the type:

$$AB + CD = AC + BD.$$

This is known as *double exchange* or *double decomposition;* it is also described as a *metathetic* reaction. The experiment is rather long, so its plan is here given as a whole:

A. To prepare zinc iodide from zinc and iodine:
   (*a*) To show that this is neither zinc nor iodine.
   (*b*) To show that it *contains* both zinc and iodine.
   (*c*) To prepare the solution of zinc iodide in water.

B. To prepare lime sulphide in solution by water from lime and sulphur.
   (*d*) To show that this *contains* sulphur as constituent.

C. To mix the solution of zinc iodide and that of lime sulphide, a chemical change taking place by which a liquid and an insoluble solid are produced.
   (*e*) To show that the liquid now *contains* lime and iodine in combination, but no zinc nor sulphur.

(*f*) To show that the insoluble solid *contains* zinc and sulphur in combination, but no iodine nor lime.

Conclusion.—Zinc iodide and lime sulphide have become zinc sulphide and lime (calcium) iodide.

18/A A. *To prepare zinc iodide.*—Weigh a clean, small evaporating dish and a short glass rod to tenths of a gram. Place in the dish 1 gram of granulated zinc and a few drops of water.

Weigh out 3.9 grams of iodine (on glass or on paper). Add about one fourth or one fifth of the iodine to the zinc in the dish, stir, and warm very slightly, just enough to start the change which itself produces much heat. When the action seems to cease, add again about the same quantity of iodine, stirring constantly, and so on until all has been used. This gives a small volume of dark-colored liquid. Holding the burner in the hand (see Appendix, 12), heat from time to time to keep the liquid gently boiling. There is some escape of purple fumes for a few seconds; this vapor is irritating, so avoid inhaling it. When the contents thicken there is a tendency to spatter; prevent this by constant stirring and moderating the heat. Finally, when the contents seem thoroughly dry, white fumes begin to escape. At this point cease heating. The product is a dry powder slightly brownish in color. When the dish is cool, weigh it with its contents. The substance weighs more than the zinc, but somewhat less than the zinc and iodine together, owing to loss by the heating.*

(*a*) *To show that this is neither zinc nor iodine:* You have seen that it shows no purple fumes upon heating, as iodine would show.

Place a small portion, such as would be taken on the point of a penknife, in a test-tube with a few drops of

* Iodine stains may generally be removed by using sodium sulphite or ammonium sulphide.

water. It dissolves as zinc would not, but with no brown color such as iodine would impart.

(*b*) *To show that it contains both zinc and iodine:* Place another small portion in a small evaporating dish, add to it a drop of nitric acid, and heat to complete dryness. You see the purple vapor of iodine, and have left in the dish zinc oxide, as seen in Exps. $15/_3$ and $16/_2$.

(*c*) Dissolve the rest of the product, zinc iodide, in 50 cubic centimeters of water (see Appendix, 14). You may see some of the original zinc unacted upon. Filter the solution (see Appendix, 13). Turn a few drops of the filtrate (that is, the liquid which has passed through the filter) into a test-tube, and add a few particles of lead nitrate (see Exp. $17/_3$) previously dissolved in water. The bright-yellow powder now appearing in the liquid is lead iodide, seen also in Exp. $15/_1$, and must be taken as further evidence of iodine as a constituent in the substance formed in 18/A. Set aside the remainder of the solution and label it *zinc iodide.* To what type of change does this formation of zinc iodide belong?

B. *To prepare calcium or lime sulphide in solution.*— 18/B
Place 5 grams, roughly weighed, of powdered lime in the larger evaporating dish. Add 100 cubic centimeters of water and boil. Touch a piece of red litmus paper to the liquid and note the effect. Add to the contents of the dish 3.2 grams, roughly weighed, of sulphur powder (flowers) and boil, stirring constantly. Keep the liquid boiling gently for a few minutes, until it becomes colored quite a deep yellow. Cease the heating, and the powder quickly settles to the bottom. Drain off the liquid as much as practicable without disturbing the sediment (this is called *decanting*), pouring the former upon a filter. Add again 100 cubic centimeters of water to the contents of the dish, boil until well colored, let it settle, and decant upon the filter. Do this four times, which will give 300 cubic centimeters or more of clear liquid. This will prob-

ably be enough, but if more is needed, it is only necessary to add another portion of water and repeat the operation. Try the action of this yellow liquid on the red litmus paper.

(*d*) *To show that sulphur is a constituent of this liquid:* Take about one half test-tubeful, add hydrochloric acid, and heat, holding at the mouth of the tube a piece of filter paper, wet with lead acetate solution. What is the effect? You also see, reappearing in the liquid, yellow, or nearly white, very finely divided sulphur.

The yellow liquid obtained in 18/B may, for present purposes, be considered as a solution in water of *lime sulphide* or *calcium sulphide*, a substance containing lime and sulphur in combination.

18/C C. *The reaction, double exchange, between zinc iodide and lime sulphide.*—Pour about one half the solution of zinc iodide, prepared in (*c*), into one of the larger beakers and drop in a piece of red litmus paper. Now, both solutions being hot, pour the lime sulphide slowly into the zinc iodide, a little at a time, constantly stirring, until the paper first turns permanently blue. Be careful to avoid any unnecessary addition of lime sulphide beyond this point. You see a white insoluble solid, a powder, form in the midst of the liquid, and settle to the bottom. A substance formed in this manner is called a *precipitate*, and the operation is *precipitation.*

(*e*) *To show that the liquid now contains lime and iodine in combination, but no zinc nor sulphur:* Let the precipitate obtained in C settle well to the bottom of the beaker; then, without disturbing it, pour off as much of the liquid as possible upon a filter. Fill up the beaker with warm water, stir the precipitate, let settle, and again decant the liquid upon the filter, collecting what runs through with the first portion. Label this, filtrate (*e*), and set it aside. Again fill the beaker with water, stir the precipitate, and set this aside to settle, labeling it, precipitate (*e*).

From filtrate (*e*) take about one half test-tubeful, and add to this a few drops of lime sulphide solution. The absence of precipitation may be taken as evidence of the absence of zinc, or perhaps, more strictly speaking, of zinc iodide.

Take again one half test-tubeful of filtrate (*e*), and add a few drops of lead nitrate solution. The bright-yellow precipitate is recognized as lead iodide, and shows the presence of iodine in combination.

Boil the rest of filtrate (*e*) to dryness in the smaller evaporating dish, having care to moderate the heat as the liquid thickens, so as to avoid spattering and possibly cracking the dish. When the last of the liquid disappears, the full heat of the flame may be applied. As a result, you see the purple vapor of iodine appear. Continue the heating as long as iodine is liberated, and you have left finally in the dish a white, chalklike powder.

That this residue is not zinc oxide, you may see by its color and by adding to the whole, or a portion of it, a very small quantity of water, boiling and testing the liquid with red litmus paper.

That this residue does not contain sulphur, you may see by adding hydrochloric acid, boiling, and testing the vapor by paper wet with lead acetate solution. In fact, it is lime, the substance with which you started.

(*f*) *To show that the white precipitate* (*e*) *contains zinc and sulphur, but no iodine nor lime:* Pour the liquid and the precipitate which was labeled precipitate (*e*) upon the filter. When the liquid has filtered through, fill the funnel with water, not quite up to the edge of the paper, and when this has passed through fill it a second time. The precipitate is thus washed free of soluble matter. Take out a portion of the wet precipitate and dry it by the heat of the flame, holding it on the spatula blade or the asbestos.

Drop some of the dried substance into a test-tube, add hydrochloric acid, boil, and test the vapor as before for hydrogen sulphide.

Drop another portion of the dried substance into a small evaporating dish, barely moisten it with nitric acid, evaporate the acid by gentle heat, then heat with the full flame. You see sulphur appear and burn, but no iodine, and you have left finally zinc oxide, seen also in Exps. $15/_3$ and $16/_2$. Boil another portion of the dried substance with a little water, and test by red litmus to see that it is not lime.

EXPLANATORY NOTE.—By the operation in 18/A you have combined zinc and iodine, making zinc iodide; in 18/B you have combined lime and sulphur, forming lime or calcium sulphide; by mixing the solutions of these two substances you have obtained an insoluble solid which contains zinc and sulphur combined—that is, zinc sulphide—and at the same time you have a liquid which contains lime and iodine combined—that is, lime or calcium iodide. Thus the two substances, zinc iodide and lime sulphide, with which you started, have exchanged constituents and produced two entirely different substances. Such a change is expressed by the general form:

$$A\,B + C\,D = A\,C + B\,D.$$

As to Notation.

19 There is used in chemistry, for the purpose of designating substances and describing changes, a system of symbols, which, at this early stage of the study, may best be regarded as a system simply of arbitrary signs, although it really has a much deeper significance. Thus the following symbols are used: For sulphur, S; for oxygen, O; for zinc, Zn; for iron, Fe (Latin, *ferrum*); for hydrogen, H; for sulphur dioxide, SO_2, signifying qualitatively that it is made of sulphur and oxygen, or contains these as sole constituents; for zinc sulphide, ZnS, and for iron sulphide, FeS, since they contain solely zinc and iron respectively, and sulphur; for hydrochloric acid, HCl, containing hydrogen and chlorine; for hydrogen sulphide or hydrosulphuric acid, H_2S, since it contains hydrogen and sulphur. The fact that sulphur and oxygen produce by chemical change sulphur dioxide is expressed in equation form thus:

$$S + O_2 = SO_2.$$

The reactions between sulphur and zinc and iron are thus expressed:

$$Zn + S = ZnS.$$
$$Fe + S = FeS.$$

The substance used under the label, hydrochloric acid, is really a solution of this substance, HCl, in water. Now, when hydrochloric acid acts on zinc sulphide, the hydrogen of the acid combines with the sulphur of the sulphide forming hydrogen sulphide, and the chlorine of the acid with the zinc of the sulphide forming zinc chloride, $ZnCl_2$; similarly in the case of the iron sulphide. These reactions are expressed by the equations: 19/1

$$ZnS + 2HCl = H_2S + ZnCl_2.$$
$$FeS + 2HCl = H_2S + FeCl_2.$$

The zinc chloride, $ZnCl_2$, and the iron chloride, $FeCl_2$, remain dissolved in the water. The numerals which you see used in this system of chemical "shorthand" may for the present best be regarded as arbitrary parts of the system. In the "additional illustrations of chemical change" (see Exp. $15/_1$, etc.) you have used lead, symbol Pb (Latin, *plumbum*), and iodine, symbol I, and lead iodide, symbol PbI_2; magnesium, symbol Mg; zinc, Zn, and oxygen, O; also the oxides of magnesium, zinc, and lead, symbols MgO, ZnO, and PbO.

What is the meaning of the following equations?

$$Pb + I_2 = PbI_2.$$
$$Mg + O = MgO.$$
$$Zn + O = ZnO.$$
$$Pb + O = PbO.$$

Referring to the experiments under substitution (see Exp. $17/_1$, etc.), you should realize the meaning of this equation, disregarding the numerals: 19/2

$$Zn + 2HCl = ZnCl_2 + H_2.$$

The symbol for sulphuric acid is $H_2[SO_4]$; that is to say, this substance contains hydrogen and something else, the nature of which need not now be considered; this "something" is represented by the symbol in the brackets. The symbol for zinc sulphate is $Zn[SO_4]$, hence the reaction between zinc and sulphuric acid is represented thus:

$$Zn + H_2[SO_4] = Zn[SO_4] + H_2.$$

The symbol for nitric acid is $H[NO_3]$, and of lead nitrate is $Pb[NO_3]_2$; hence the reaction between lead and nitric acid is represented thus:

$$Pb + 2H[NO_3] = Pb[NO_3]_2 + H_2.$$

Note that these last three equations fall under the general form

$$A + B\,C = A\,C + B.$$

4. Additional Illustrations of Physical Properties

20 **Distillation.**—See Exp. $24/_5$, and Appendix, 18, and Part I, No. 12.

20/1 **Sublimation.**—Heat gently a small fragment of iodine in a dry test-tube. Describe the phenomenon. Note the color of the vapor and its odor (cautiously), and its weight compared with that of air (invert the tube). Does the solid melt? Does the vapor pass through the liquid to the solid form on cooling? Is the solid which is deposited (the sublimate) crystalline or not? (See Part I, No. 12.)

21 **Solution and crystallization.**—Add a few drops of hydrochloric acid to a few drops of a solution of lead acetate in a test-tube. What takes place? (Precipitation.) Boil the contents of the test-tube, adding a little water, if necessary, to dissolve the white powder first formed; set aside to cool. What takes place on cooling?

EXPLANATORY NOTE.—The white powder formed on mixing the two liquids is lead chloride (symbol, $PbCl_2$). Does its formation involve a physical change or a chemical change? It is not soluble, or only slightly so, in cold water; hence it appears as a solid, a powder, in the midst of the liquid. Lead chloride dissolves in hot water, and, as the solution cools, reappears in crystalline form; this is a purely physical change.

21/4 **Solution and crystallization.**—Dissolve about 10 grams of alum (a porcelain evaporating dish is convenient) in 50 cubic centimeters of hot water. Filter the solution. Dissolve about twice as much copper sulphate in a similar manner. Filter this. Mix the two solutions; boil down to about one half the volume, and set aside to crystallize (it may be left until the next day). Can you distinguish the two substances in the crystals? Can you distinguish them in the solution?

21/7 **Water of crystallization.**—Put about 1 gram of copper sulphate crystals in a dry test-tube, and weigh carefully the tube and contents; to tenths is sufficient. Heat slowly, holding the tube so that the open end is a little the lower.

What condenses on the cooler portion of the tube? When the liquid has disappeared let the tube cool, and weigh again. Shake out the white *dehydrated* substance, and let a drop of water from the finger come in contact with a portion of it. Observe carefully what takes place. Bring a drop or two of alcohol in contact with another portion. The dehydrated, *amorphous* substance may be redissolved with hot water, and crystallized as it was at first. (See Part I, No. $21/_6$.)

Heat in similar manner some alum crystals, also some crystals of potassium dichromate, or of potassium nitrate.

Efflorescence and deliquescence.—Expose to the air for 21/8
twenty-four hours, more or less, (*a*) some crystals of sodium carbonate or of sodium phosphate; (*b*) some calcium chloride or sodium hydroxide (see also Exp. $17/_1$ b).

Heat of solution.—Determine the effect on temperature 22
(see Appendix, 15) of dissolving in water (*a*) some common salt—that is, sodium chloride (use a beaker half filled with water and about a tablespoonful of salt); (*b*) some sodium hydroxide; (*c*) some hydrochloric acid (concentrated).

Melting point, determination of (see Appendix, 16).—De- 23
termine the melting point and freezing point of paraffin thus: Prepare a tube of thin wall and small bore by heating in the flame a piece of glass tubing, an inch or two from its open end, until the glass is well softened; then, drawing it out slowly until the bore becomes quite small, and finally applying the flame at the narrow part and separating completely the two portions of the tube. The narrow end is thus closed by fusion. Put into this a few bits of paraffin; warm just enough to melt the latter; shake or jar the tube so that the liquid shall completely fill the narrow portion, leaving no air bubble. With a rubber band attach the tube alongside the thermometer so that the paraffin is about opposite the bulb. Suspend the thermometer and tube over a beaker of water which is supported on an asbes-

tos board or a wire gauze, so that the bulb and paraffin shall be well immersed. Slowly heat the water and observe the temperature when liquefaction is first noted. When this is complete, let cool, and observe the temperature when solidification begins. Repeat four or five times, until the several readings of each point are fairly concordant. As the melting or the freezing point is approached the temperature should be allowed to change *very slowly*, and the water should be well stirred. The freezing point may be somewhat lower than the melting point, due to *surfusion*.

24 **Boiling point, determination of, and conditions affecting.**—Determine the boiling point of water thus (see Appendix, 17): Use a distilling flask of about 200 or 225 cubic centimeters capacity, with a side delivery tube carrying a short piece of rubber hose. Half fill with water. Through the stopper insert a thermometer (wetting it first) so that the bulb is below the exit and above the surface of the water, high enough to avoid being spattered during boiling. Support the whole on asbestos, or gauze, and the iron stand so that there may be no danger of overturning. Heat until the liquid boils and the temperature reaches its maximum, which is steadily maintained for five minutes or more. This is the *boiling point*. It should be, with a good thermometer, 100° C.; but thermometers are often inaccurate, and may be corrected by actual observation, made with due care.

Vary the experiment as follows:

24/1 1. By pinching with the tongs, close the rubber exit tube while the liquid is still boiling, and note the effect on temperature. Let it rise only about 2°, then open and observe what takes place.

24/2 2. Lower the thermometer until the bulb is immersed, boil, and note again the maximum temperature, first with the tube open, then with it closed. The temperature of the boiling liquid is likely to be higher than that of the vapor under similar conditions.

3. Let it cool sufficiently, then add to the water about a tablespoonful of salt and a few crystals of copper sulphate, boil, and observe the temperature of the vapor as before (it should be unchanged). 24/3

4. Lower the thermometer and take the temperature of the boiling solution. 24/4

Collect some of the liquid (*distillate*) which drops from the exit tube. Does it show any color? Has it the taste of salt? (*Distillation.*) 24/5

Fit a test-tube well with a cork, fill about one third with water, and boil. After it has boiled a few seconds and while it is still boiling, remove from the flame, quickly insert the cork, invert the tube, immerse the stoppered end just under the surface of some water in a convenient vessel, and pour cool water on the upper end. What takes place? What is the explanation of the phenomenon? Let cool, uncork, and note the inrush of air. If the cork does not come out easily, force a pin between the cork and the glass and withdraw it. 24/1 a

Optional experiment.—In a small evaporating dish evaporate to dryness some distilled water. Is there any residue? 25

Similarly evaporate to dryness some sample of natural water. Is there a residue?

CHAPTER II

EXPERIMENTS ILLUSTRATING THE FUNDAMENTAL QUANTITATIVE LAWS OF CHEMICAL CHANGE

33 NOTE.—Study with special care the quantitative relations in the experiments of this chapter. In all the measurements of quantity which you make, whether in this work or in the work of subsequent chapters, try to make an estimate of the uncertainty which is necessarily involved in the measurements by reason of the conditions in which you work. In some of the problems it will be important to make at least two measurements of the same quantity, in order to show how much uncertainty is thus involved. As the work advances, after you have studied the primary topic which is illustrated, give some thought to the topics already passed, which may find secondary illustration by the experiment in hand, especially as to these general laws, and also as to the kind of reaction involved, and other definitions presented in Chapter I.

Have care always to state in your notes the specific topic to be illustrated, and, when practicable, the specific experimental problem to be solved; then describe your method of solution, endeavoring to give the essential features, apparatus, incidental observations, etc.; then, as a rule, present the data of observation, carefully labeled, the calculations, if any, and the final result, with your conclusions therefrom.

In recording data, be sure that your notes include all original observations. For example, you are to determine the weight of a substance contained in a dish; you do so by determining the weight of the dish with its contents, and the weight of the dish alone, the difference between these being the weight sought. Now, the important point is to record in your notes, not simply this difference, but the original weights which by subtraction give the desired value.

Notes should be written in the laboratory, during the progress of the experiment, and quantitative data should be recorded *at once*. Do not rush through an experiment and then try to write up your notes from memory, perhaps outside the laboratory. Do not delay recording results until you have learned whether they are good. Put them all

down, and later mark "erroneous," if necessary. Do not be discouraged if the result at first trial is unsatisfactory. You will often need to perform an experiment once to learn how to do it, and need to repeat for successful results. It is well to bear in mind that practical chemistry is more or less a handicraft, and that you will surely fail to get good return for your time and labor unless experiments are performed carefully as well as studiously.

Make it a rule to read through the directions for the *whole* experiment before you start upon its performance.

1. The Law of Persistence of Mass

A. **Preliminary.**—Some apparent contradictions:

(*a*) Place about 1 gram of mercury sulphocyanate in a small evaporating dish, counterpoise the dish and its contents on the balance, ignite the substance by a match or a hot iron wire. Describe the phenomenon, and note the effect on the equilibrium of the balance. Had you made the experiment without using the balance, what would you have inferred as to the effect of the change on the quantity of substance? 34/1

(*b*) *An experiment for the teacher to perform before the class.*—Burn a taper, or alcohol in a small lamp, in such manner as to collect and weigh the products of combustion. This may be conveniently done as follows: Suspend the taper or very small lamp (which can be made from a short test-tube, a cork, and a piece of wicking) from the lower end of a student-lamp chimney, to the upper end of which is fitted a perforated cork carrying a train of four absorption tubes; the first two of these are filled with fragments of sodium hydroxide, and the last two with fragments of calcium chloride; to the farther end of the train is attached a rubber hose leading to a filter pump or to an aspirator; the whole apparatus—lamp, chimney, and absorbing train—is then suspended from the arm of a suitable balance and counterpoised. When this is ready, start the pump which draws a current of air through the apparatus, 34/2

carrying with it the products of combustion into the tubes, where they are retained. Light the lamp and allow it to burn fifteen or twenty minutes, noting the effect on the

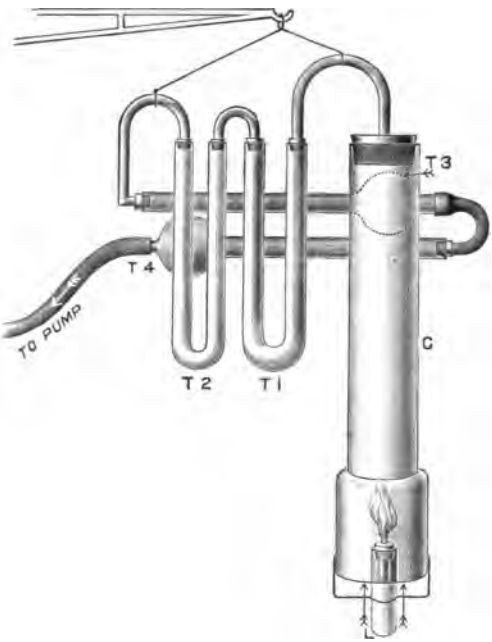

FIG. 1.—Diagram of apparatus for Exp. 34/2. *L*, alcohol lamp; *C*, lamp chimney; T_1, T_2, T_3, and T_4, absorption tubes.

equilibrium of the balance. How does the weight of the products of combustion compare with the weight of the material burned? (Fig. 1.)

34/3 *An alternative form* of apparatus for this experiment may be more simply provided as follows: Make a cylinder of wire gauze which shall fit closely in the upper half of the student-lamp chimney; fit the lower end with a cork, through which several holes are bored, and to which a short piece of taper may be attached (see Fig. 2), or, preferably,

suspend the little alcohol lamp as in Fig. 1. Fill the gauze cylinder with fragments of sodium hydroxide, and suspend the whole by a loop of wire from the arm of the balance. Light the lamp or the taper. Although the products of combustion are not so fully retained as in the preceding form, the gain in weight is made evident by burning for a few minutes.

FIG. 2.

EXPLANATORY NOTE.—The alcohol and the candle contain carbon and hydrogen as elementary constituents. These in the process of burning combine chemically with the oxygen of the air, forming as products gaseous carbon dioxide and hydrogen oxide—i. e., water. These products are carried into the tubes, where the carbon dioxide is retained by the sodium hydroxide, and the water is retained partly by condensing to liquid form, and partly by the strongly hygroscopic substance, calcium chloride. The products of the combustion, therefore, weigh more than the material burned by just the weight of the oxygen taken into combination from the air. 34/4

All the ordinary fuels likewise contain carbon and hydrogen; therefore the products of ordinary combustion are carbon dioxide and water, and combustion itself is simply an instance of chemical action accompanied by heat and light. (Compare with Exps. 13, $15/_2$ and $15/_3$.)

B. To illustrate the law:

(1) Recall or repeat Exps. $13/_{2a}$, and $15/_1$.

(2) In a gas generator (see Appendix, 6) fitted with a thistle-tube and a delivery tube place a charge of marble (i. e., calcium carbonate, symbol $CaCO_3$) in small lumps, with enough water to seal the end of the thistle-tube; in another vessel (a bottle or a deep beaker) place a strong solution of sodium hydroxide; in a third (a small beaker), some concentrated hydrochloric acid and a glass rod. Counterpoise the three vessels with their contents on the heavier balance (see Appendix, 1, A). Cautiously turn the acid, a few drops at a time, into the generator (observe 34/5

what takes place), letting the reaction go on slowly through a measured interval of time while the delivery tube opens into the air, and until the effect on the equilibrium is unmistakable. (What is the effect?) Then drop the delivery tube into the second vessel so that the gas bubbles through the solution; again counterpoise, and continue the reaction at about the same rate, and during the same length of time as before; then again note the effect on the equilibrium.

85 **Secondary observation.**—To show that an invisible gas, quite different from air and not before present, is produced by this change, remove the material from the balance, drop the delivery tube into a clean beaker or bottle, partly covered by a glass plate (see Appendix, 19, V), and pour a little more acid into the generator. After the effervescence has continued a few seconds, slide the plate to one side and plunge a lighted match into the bottle. The gas produced is *carbon dioxide*, symbol CO_2.

86 Explanatory Note.—This experiment, owing to the limitations in weighing, can give but crude quantitative results, showing simply that the balance, which gives no indication of differences less than 0.2 or 0.3 of a gram, indicates unmistakable loss of weight in the first conditions, but no loss in the second, although the reaction goes on as in the first.

The chemical name of marble is calcium carbonate, symbol $CaCO_3$. It is a salt derived from the metal, calcium, and carbonic acid, symbol H_2CO_3. By the action of hydrochloric acid on this substance, calcium chloride, another salt, symbol $CaCl_2$, is produced, and carbonic acid. The latter substance breaks up at once into carbon dioxide, symbol CO_2, and water, symbol H_2O. The carbon dioxide passes into the atmosphere as an invisible gas, the water adds itself to the rest of the water, and the calcium chloride remains in solution. These two reactions are expressed in equation form as follows:

$$(1)\ CaCO_3 + 2HCl = CaCl_2 + H_2CO_3.$$
$$(2)\ H_2CO_3 = CO_2 + H_2O.$$

Sodium hydroxide, symbol NaOH, is a base (see Part I, 30/2), and when the carbon dioxide comes in contact with its solution in the sec-

ond bottle the two substances combine and another salt is produced, namely, sodium carbonate, symbol Na_2CO_3. This remains in solution. The following equation expresses this reaction:

$$2NaOH + CO_2 = Na_2CO_3 + H_2O.$$

2. The Law of Fixed or Definite Proportions

(*a*) Evaporate to dryness a few drops of hydrochloric 37
acid solution in the small evaporating dish, heating gently with the caution necessary in this operation (see Appendix 12). Acid of a good degree of purity is needed.

(*b*) Likewise evaporate a few drops of ammonium hydroxide solution. Describe the results.

(1) Carefully weigh to tenths of a gram the small porcelain dish with a short glass rod, both articles being clean and dry.

[It will be advantageous to have two or even three weighed dishes to use. They may be distinguished by scratches on their edges made with a file.]

With some suitable measure (a test-tube with a slip of gummed paper or a file scratch on its side will serve, although a burette or graduated pipette is better) take two equal volumes of the ammonium hydroxide solution, pouring them into a weighed dish. Exactly neutralize this liquid with the hydrochloric acid solution, using litmus paper as indicator, and adding the acid drop by drop, stirring after each addition, until the paper just turns permanently pink. Note carefully the volume of acid used. Evaporate the contents of the dish to dryness on the water-bath; this is facilitated by frequent stirring toward the end of the operation. Let cool, and determine the weight of the salt obtained. How will you make sure that the salt is completely dried?

Redissolve the salt, using very little water; filter if necessary, and crystallize. Dry the crystals on filter paper, and test them with blue and with red litmus paper slightly wet.

(2) Take the same volume of hydrochloric acid which was used in (1), add to it only one measureful of ammonium hydroxide, evaporate, and weigh as before the salt obtained. Redissolve, filter, and crystallize this salt. Test the crystals with red and with blue litmus paper.

(3) Take again the same volume of acid as previously used, add to it three measurefuls of ammonium hydroxide, evaporate, and weigh as before.

Redissolve, filter, and crystallize this salt. Test the crystals with red and with blue litmus paper.

38 QUESTIONS.—Are the three portions of salt thus obtained different samples of the same substance, or are they different substances? What is the ratio between the quantities of salt obtained in the three cases? Have you any evidence that hydrochloric acid or ammonium hydroxide passes off during the evaporation, in (1)? in (2)? in (3)? What is the logic of the experiment? Reason it out fully. Is heat liberated when hydrochloric acid and ammonium hydroxide are mixed?

39 EXPLANATORY NOTE.—The salt obtained by the combination of hydrochloric acid and ammonium hydroxide (symbol NH_4OH, a base) is named ammonium chloride, symbol NH_4Cl, and the reaction is thus expressed: Hydrochloric acid and ammonium hydroxide produce ammonium chloride and water; or in equation form,

$$HCl + NH_4OH = NH_4Cl + H_2O.$$

3. The Law of Multiple Proportions

40/a (*a*) Take of iodine and of mercury in the ratio of 252 parts by weight of the former and 199 of the latter; for the actual experiment weigh out accurately (in some glass vessel, as both substances attack metals) 6.30 grams of iodine, and 5.00 grams of mercury. Transfer the mercury to a mortar (previously weighed with its pestle on the heavier balance). Add a few drops of alcohol, then a small portion of the iodine, and rub gently with the pestle; then add another portion of iodine, and rub; and so on until all the iodine is used and the whole is thoroughly mixed.

It is well to keep the mixture slightly moist with alcohol during only the first stages of the operation, in order to avoid overheating, otherwise there may occur a slight flash in the mortar, accompanied by the fusion of the substance. If this happens, it is better to clean the mortar (use sand to scour), and start the experiment anew. Avoid inhaling the fumes of iodine, and protect the hand, if desired, with a fold of the towel. Iodine stains may be removed with sodium sulphite or ammonium sulphide.

Persistent rubbing may be necessary even after the substances seem well mixed. The operation when completed should yield a bright-red powder, mercuric iodide, symbol HgI_2.

Weigh the mortar and contents on the heavier balance.

Take out a small sample and apply alcohol to it in a test-tube. [The alcohol should not be colored by the iodine; if it is so colored, continue the rubbing.] Warm the alcohol to gentle boiling, having care that the alcohol vapor is not ignited by the flame; set aside to cool. The amorphous powder dissolves in hot alcohol, and deposits on cooling in crystalline scales, sometimes bright scarlet, sometimes yellow (allotropic forms). 40/1

Sublime another small portion of the red powder in a dry test-tube, note the red and yellow sublimate (allotropic forms); rub the yellow with a glass rod. Note the crystalline residue after fusion. 40/2

(*b*) Take 5.60 grams of mercuric iodide obtained in (*a*), which is equal to $\frac{1}{2}$ (6.3 + 5) and 2.50 grams of mercury. Add the mercury in small portions at a time to the mercuric iodide in a mortar, rubbing thoroughly after each addition. This should yield a greenish-yellow powder (mercurous iodide, symbol HgI). If the color is not satisfactory, continue the rubbing and, perhaps, allow to stand for twenty-four hours. 40/b

Weigh the mortar and contents on the heavier balance.

Treat a small portion as in (*a*) with boiling alcohol. Does it dissolve? 40/3

40/4 Sublime another portion; note the character of the sublimate and the residue after heating.

40/5 EXPLANATORY NOTE.—Mercurous iodide is insoluble in hot alcohol and decomposes on heating into mercuric iodide and mercury.

QUESTIONS.—How do you differentiate between mercury and iodine mixed, and mercuric iodide? between mercuric iodide and mercurous iodide? between mercuric iodide and mercury mixed, and mercurous iodide? How does the weight of the mercuric iodide obtained compare with that of the mercury and iodine used? What becomes of the alcohol? How does the weight of the mercurous iodide compare with that of the mercuric iodide and the mercury used? What is the ratio between the quantities of mercury in the two substances reckoned for constant quantity of iodine? between the quantities of iodine, reckoned for constant quantity of mercury? What is the logic of the experiment? Show clearly that it is not contradictory to Law 2. What does Law 2 affirm in regard to these two reactions? What does Law 1 affirm with regard to them? Is heat liberated in the reaction between mercury and iodine?

4. The Law of Equivalent Proportions*

41 **General problem.**—To investigate the relation between those quantities of different substances which produce equal chemical effects.

Specific illustrative problem.—A. To determine the relation between the quantities of oxygen which combine respectively (1) with 2.40 grams of magnesium, and (2) with 6.50 grams of zinc.

B. To determine the relation between the quantities of hydrogen liberated from hydrochloric acid respectively (3) by 2.40 grams of magnesium, and (4) by 6.50 grams of zinc.

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* Inasmuch as the experiment under this topic involves the measurement of gas-volume, the instructor may prefer to introduce the laws of Boyle and of Charles, Chapter IV, at this point. The writer prefers to give them simply as arbitrary rules and to study them later, rather than to interrupt the logical development of this chapter.

A 1

To determine the quantity of oxygen which combines with 2.40 grams of magnesium: Recall Exp. 15/2. Weigh to hundredths of a gram a porcelain crucible with its lid, clean and dry. Weigh out exactly 1.00 gram of magnesium ribbon. Breaking this into small pieces, transfer the whole to the crucible. Support the latter, covered by its lid, on the pipe-stem triangle and iron stand (see Appendix, 11). Apply heat, slowly at first, then the full heat of the flame. Raise the lid slightly from time to time to see what is taking place and to let air in, but avoid losing any of the white smoke. When the danger of this is passed, remove the lid and continue the heating about fifteen minutes. When the crucible is cool, weigh it with its lid and contents to hundredths. To make sure that the reaction is complete, heat, cool, and weigh again. When heated, the magnesium combines with the oxygen of the air and the product is magnesium oxide, MgO. Calculate from the mass of oxygen which combines with 1.00 gram of magnesium what mass combines with 2.40 grams of the same. (Upon what law is this calculation based?) At least two determinations of this value should be made. 41/1

Secondary items.—Describe the phenomenon seen and the substance produced. Does the reaction evolve heat? Does magnesium oxide dissolve in hot water? Test with red litmus paper. Does it dissolve in dilute sulphuric acid? Boil it in the crucible or dish with a very little of the dilute acid, filter, and crystallize the salt, magnesium sulphate, $MgSO_4$ (compare Exp. 17/2). How does the reaction between magnesium oxide and sulphuric acid, symbol H_2SO_4, differ from that between magnesium and the same acid? 41/a

An Alternative Method

This method involves making, first, magnesium iodide, MgI_2, then heating this substance, by which it is decomposed into iodine and mag- 41/b

nesium, and the latter is converted into the oxide, MgO, all without loss of substance other than the iodine. The final result is the same as in 41/1, but it is brought about with less elevation of temperature.

Weigh to hundredths a small evaporating dish with a short glass rod. Weigh out carefully 0.50 of a gram of magnesium, and place this in the dish. Weigh out about 5 grams of iodine. Pour on the magnesium enough alcohol to cover it. Then add the iodine, a little at a time, stirring constantly. With a little care the reaction may be so controlled that the heat evolved shall not cause spattering and consequent loss of material. When the magnesium is entirely acted upon, heat the dish and contents on the water-bath until only a thick, siruplike mixture is left. Then transfer the dish to the iron ring and heat with a very small flame, holding the burner in the hand (as before directed for careful evaporation; see Appendix, 12), and stirring constantly. Continue this until the danger of spattering is past, then apply the full heat of the flame. As this causes iodine fumes, it may be necessary to perform this part of the experiment in the hood. Continue the heating for a few minutes after the iodine has entirely disappeared. Finally let cool, and weigh the dish and content, which is magnesium oxide, a fine powder only slightly brown in tint. The calculations are the same as in the first method.

A 2

41/2 To determine the quantity of oxygen which combines with 6.50 grams of zinc:

It is impracticable to do this by direct combustion, as in the magnesium experiment. The method is to act on zinc with nitric acid, produce zinc nitrate, dry this, then ignite it and thus produce zinc oxide without loss of other than volatile material. Refer to Exps. 15/2 and 16/2.

Weigh a small evaporating dish with a short glass rod to hundredths of a gram. Weigh out 2.00 grams of granulated zinc. Transfer the latter to the dish. Pour a drop

or two of nitric acid down the rod, letting it come slowly in contact with the zinc. (Remember that nitric acid is very corrosive.) When the first violent reaction is over, add a few drops more of acid, and so on until the zinc is completely dissolved with the least acid that will serve. Evaporate the contents of the dish to dryness on the water-bath. The evaporation may be performed more quickly, but only with closer attention, by holding the burner in the hand, having the flame low, and applying just enough heat to keep the liquid gently boiling, until there is danger of spattering, and then not enough heat to cause the formation of bubbles (see Appendix, 12). When the salt is thoroughly dry, increase the heat, and finally give it the full flame for about fifteen minutes. When the reaction is finished, let the dish cool, and weigh it with its contents to hundredths of a gram. The product is zinc oxide. To make sure that the reaction is complete, heat, cool, and weigh again. From the mass of oxygen which combines with 2.00 grams of zinc, calculate what mass combines with 6.50 grams of the same. At least two determinations of this value should be made.

Secondary items.—Describe the reaction between zinc and nitric acid. Is heat evolved? What takes place when zinc nitrate is heated? Does zinc oxide dissolve in water? in dilute sulphuric acid? Optional experiment: Filter, and crystallize the zinc sulphate (see Exp. 17/2). 41/c

How does the reaction between zinc oxide and sulphuric acid differ from that between zinc and the same acid? What is obtained by the action of hydrochloric acid on zinc oxide?

Summary.—Compare with each other the individual quantitative results with magnesium, also those with zinc; then compare the average of the first with the average of the second. What is the conclusion as to the masses of oxygen which combine respectively with 2.40 grams of magnesium and with 6.50 grams of zinc? 41/d

B 3

41/3 To measure by volume and calculate into weight the quantity of hydrogen liberated from hydrochloric acid by 2.40 grams of magnesium:

(*a*) To measure the capacity of the collecting bottle, No. x (a bottle with glass stopper holding about 2,500 c.c.): Fill the tank with water, which should be brought to the temperature of the room, if necessary by heating portions in the iron vessel. Fill the collecting bottle completely and put the stopper in place. Measure the volume of this water in cubic centimeters; this may be done by pouring, without loss, into a graduated cylinder, or, if such vessel is lacking, as follows: Fill a graduated flask repeatedly from the bottle (one holding 750 c.c. is convenient), and weigh, in a vessel not unnecessarily large or heavy, the last residue which is insufficient again to fill the flask; the weight of the water in grams may be taken with only inconsiderable error as its volume in cubic centimeters. Determine thus the capacity at least twice. The variations should not be more than one or two cubic centimeters.

[Why not weigh at once the bottle filled with water?]

41/3b (*b*) To generate the gas: Weigh out carefully 2.40 grams of magnesium ribbon, transfer without loss to the gas generator (see Appendix, 6). Add water just sufficient to seal the thistle-tube; test for leakage by blowing in the exit end of the delivery tube until the water rises four or five inches in the thistle-tube, then pinching the rubber hose, or closing the end of the tube with the tongue. The column of water should maintain its height, or sink only slowly. A film of water between stopper and glass will help to prevent leakage, or, as a last resort, the stopper may be sealed with vaseline or with paraffin.

Have some strong hydrochloric acid in a beaker. It will be found convenient to let a test-tube lie in the bulb of the thistle-tube; it may be used like a rod to aid in

pouring from the beaker, and the rounded end makes a kind of valve.

(*c*) To collect and measure the gas: Fill the collecting 41/3c
bottle with water, put in the stopper, invert in the tank, remove the stopper, incline the bottle so that it rests on the wall of the tank and slip into its mouth the bent delivery tube (compare Appendix, 19, I). Pour acid, a little at a time, into the thistle-tube until no more gas is generated. As much heat is liberated in this reaction, it is well to set the generator down into the water of the tank. Ascertain now or later the volume in cubic centimeters of the acid turned in.

When the effervescence ceases, raise the bottle so that the water within and without is as nearly as practicable at the same level, withdraw the delivery tube, insert the stopper under water, and remove the bottle from the tank. Measure the volume of the water left in the bottle, and calculate the volume of the gas collected.

Observe the temperature of the water in the tank, which should be that of the room, so that it may be safely assumed that the temperature of the gas is the same.

Observe by the barometer the pressure of the atmosphere (see Appendix, No. 15 B).

Take note *that hydrogen with air may make a dangerously explosive mixture; therefore have great care that the hydrogen is not brought near a flame.*

Calculations. (See Appendix, 20.)—The data of this 41/3d
problem are:

The weight of metal used;

The total capacity of the bottle;

The volume of acid turned in (how is this used?);

The volume of water left in the bottle;

The temperature and pressure of the gas.

From these is first calculated the volume of the gas generated, measured in cubic centimeters at the observed temperature and pressure.

Now, since change of temperature and change of pressure cause change in the volume of gases, it is important that observed volumes, in order to be comparable, should be reduced to standard conditions of temperature and pressure. It is customary to use as standards 0° C. for temperature and 760 millimeters of mercury column in the barometer for pressure. The method of reducing observations to the standards may be taken arbitrarily now, to be explained in Chapter IV. The observed volume is reduced to volume at 0° by applying the law that "a volume of gas at 0° increases by $\frac{1}{273}$ of itself for each degree of rise in temperature, pressure remaining unchanged." The observed volume is reduced to volume at 760 mm. by multiplying by the observed pressure in millimeters and dividing by 760; but since the gas is collected and measured over water, it is saturated with water vapor, and the observed pressure is made up of the true pressure of the gas plus the pressure of the vapor at the given temperature. The pressure or tension of the water vapor at different temperatures is obtained by observation, and given in tables accessible in books of reference; this value for the ordinary temperature of 20° is 17 mm., and may be subtracted from the observed pressure as a slight correction. (See Appendix 22.)

The volume of gas generated, thus brought to standard conditions, is finally calculated into weight, by aid of the fact that one liter of hydrogen at 0° and 760 mm. weighs 0.0899 of a gram. Does the fact that the hydrogen collected in the bottle is mixed with air coming from the generator affect the problem?

At least two fairly concordant determinations should be made of the hydrogen by magnesium.

B 4

41/4 To measure by volume and calculate into weight the quantity of hydrogen liberated by 6.50 grams of zinc (compare Exp. $17/_1$): Proceed as in B 3, (*b*) and (*c*), using 6.50

grams of granulated zinc. At least two fairly concordant determinations of this value should be made.

Summary.—Compare with each other the individual 41/5
results from magnesium; also those from zinc; then the average of the first with the average of the second. What is the conclusion as to the quantities of hydrogen liberated respectively by 2.40 grams of magnesium and by 6.50 grams of zinc? Compare the results in A and B. Are the masses of magnesium and zinc which produce equal chemical effect in combining with oxygen also the masses which produce equal effect in liberating hydrogen?

5. The Law of Gas-volumetric Proportions

General problem.—To determine the proportions by volume in which gaseous substances react.

Specific illustrative problem.—To investigate the propor- 47
tion by volume in which nitrogen dioxide reacts (1) with air and (2) with oxygen.

EXPLANATORY NOTE.—The plan of work under this topic must be to state first the fundamental fact involved, and to let the experiment serve rather to illustrate the practical application of this fact to the observations in hand.

Nitrogen dioxide, symbol NO (named also nitric oxide), combines with oxygen on contact in the ratio of 4 volumes (gaseous) of the former to 2 volumes (gaseous) of the latter, forming 2 volumes (gaseous) of *nitrogen tetroxide*, symbol N_2O_4, a substance which is soluble in water. Air is a mixture of oxygen and nitrogen, 1 volume of the former to 4 volumes of the latter. Nitrogen does not react with nitrogen dioxide.

(*a*) To calibrate two gasometric (i. e., gas-measuring) 47/a
tubes: Use two test-tubes, 230 mm. (9 in.) long and 18 mm. ($\frac{3}{4}$ in.) wide, and as a unit tube one about 44 mm. ($1\frac{3}{4}$ in.) long and 10 mm. ($\frac{3}{8}$ in.) wide. Divide the large tubes into portions of equal capacity by filling the small tube with water, pouring its content into the large tube, and marking the level of the water with a rubber band.

47/b (*b*) To prepare nitrogen dioxide: Put into the generator a charge (to about half fill) of *ferrous sulphate* (commercially known also as copperas and as green vitriol) in small lumps, with water just enough to seal the thistle-tube. Have strong nitric acid with a rod in a small beaker. Turn the acid a little at a time into the generator. Collect the gas over water in the small collecting bottles (see Appendix, 19, I), rejecting, as probably impure, the first three or four bottlefuls; then retain for use stored in the bottles. *Carefully avoid inhaling the gas; also handle the acid with care, as it is very corrosive.*

47/1 (1) By means of the gasometric tubes, measure out volumes of air and of nitrogen dioxide in the ratio of 5 : 2; pouring the latter under the water of the tank, upward from the bottle into the tube (see Appendix, 19, II). It is advantageous to use large rather than small quantities.

Pass the content of one tube, under water, into the other (what takes place?), and, after sufficient interval, read the volume of the residual gas. Repeat the operation enough to get fairly concordant results. If the gas is pure and the measurements well made, the result is 4 volumes in proportion to the 5 and the 2 which were taken; therefore 3 volumes, or $\frac{3}{7}$ of the mixture, disappear in the reaction. (What is the cause of this contraction?)

Take a measured sample of the residual gas, add to this a measured volume of air, and observe the contraction; if it is not zero, make repeated additions, until there is no further contraction.

Take another measured sample of the residual gas, add to it a measured volume of nitrogen dioxide; if the contraction is not zero, continue the additions until it is.

Does the first residual gas contain oxygen? Does it contain nitrogen dioxide? What is it?

47/2 (2) Measure out, as in (1), volumes of oxygen and of nitrogen dioxide in the ratio of 1 : 2. Mix them and measure the residual volume and the contraction.

If there is a residual gas, test it for further contraction by repeated additions of oxygen. What is indicated here by contraction or absence of contraction?

Likewise test the residual gas for further contraction by additions of nitrogen dioxide. What does contraction signify here? Why are observations of temperature and pressure unnecessary? (For a convenient form of record see Prob. 8, No. $65/_6$, Part I.)

APPLICATIONS.—Measurement of contraction, in conditions like those just indicated, may be used to determine the real volume of oxygen or of nitrogen dioxide in a given sample—that is, the purity of the sample—provided only that there is nothing present to cause contraction in the given conditions, except these two substances. It is only necessary to make sure that the substance to be measured has been entirely used in making the observed contractions. (How is this made sure?) Then the volume of oxygen actually present may be obtained by the proportion 3 : 1 :: observed contraction : volume of oxygen (why?); and the volume of nitrogen dioxide may be obtained by the proportion 3 : 2 :: observed contraction : volume of nitrogen dioxide (why?). Apply this to determine the percentage purity of your sample of nitrogen dioxide and of oxygen. Your sample of the first will be of good quality if you reject enough to have it free from nitrogen coming from the air. Commercial oxygen is likely to be impure, also, from the presence of nitrogen. 47/3

EXPLANATORY NOTE.—The reaction by which the nitrogen dioxide is produced is rather complicated for complete explanation at this stage; the following may suffice: The proximate (see No. 26, Part I) constituents of nitric acid are water and nitrogen pentoxide; the latter very readily gives up a portion of its oxygen to other substances, and becomes a lower oxide of nitrogen (example of multiple proportions), in this instance the dioxide; the agent which takes this oxygen from the nitrogen pentoxide is ferrous oxide, a proximate constituent of the ferrous sulphate; in thus taking oxygen this substance is converted into a higher oxide of iron—namely, ferric oxide (example of multiple proportions). 47/4

Substances which thus give up the whole or a part of their oxygen are called *oxidizing agents;* those which thus take on oxygen are called *reducing agents.* 48

6. The Law of Persistence or Conservation of Energy applied to Chemical Phenomena

Heat Disturbance in Chemical Reactions

(The student should read Nos. 50–55, Part I, before undertaking the experiment.)

50/1 THE UNIT OF HEAT.—The unit of quantity which is used in measuring heat is defined as the quantity of heat which raises 1 gram of water 1° in temperature, or, strictly defined, from 0° to 1° C., but, as more commonly used, from 18° to 19°. This unit is named the *calorie* (or *gram-calorie*). For convenience, a unit equal to 1,000 calories is often used, the *kilogram-calorie*, designated by the abbreviation, Cal., while for the smaller unit the abbreviation, cal., is used.

50/2 The quantity of heat disturbance is most simply measured when the reaction takes place quickly and in water solution, as in the neutralization of bases and acids. The result is commonly reckoned for the combining weights in grams of the substances used. (Recall in what reactions you have noted the liberation of heat.)

50/3 **Illustrative problem.**—To determine in calories the quantity of heat liberated in neutralizing 125 grams (its combining weight) of *oxalic* acid with ammonium hydroxide, both being in water solution.

Weigh out 5.00 grams of oxalic acid; dissolve this in 100 c.c. of water in a beaker. Take sufficient ammonium hydroxide solution to neutralize this acid,* and add enough water to bring its volume up to 100 c. c. Have the two solutions in beakers, and at a temperature not far different from that of the room. Observe carefully the temperature of each solution. Quickly pour one into the other, stir with the thermometer, and observe the resultant temperature. (The salt, ammonium oxalate, may be readily crystallized from this solution by slight concentration.)

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* The quantity will depend on the strength of the solution. It is recommended that the instructor determine this for the class, or assign it to some student as a problem.

Calculations.—In dilute solutions it may be assumed, for calculations such as these, that the quantity of heat needed to raise the solution one degree is the same as that needed to raise the water which it contains one degree. Sometimes the weight of the water is taken, and sometimes the volume of the solution is reckoned as if it were pure water. The latter method is somewhat the simpler. In this experiment, the two volumes being equal, the temperature of the mixture would be the mean of the temperatures before mixing, if no heat were liberated. The difference between this mean and the observed resultant temperature is the number of degrees that the 200 c.c., reckoned as so many cubic centimeters or grams of pure water, have been raised by the heat of the reaction. Calculate from this the calories of heat which would be liberated by neutralizing 125 grams of oxalic acid in dilute solution. Some heat is of course taken up by the material of the containing vessel; how much, it would be necessary to determine, if greater accuracy were demanded. **50/4**

At least two determinations should be made of this *heat of neutralization.*

CHAPTER III

COMBINING WEIGHTS—NOTATION—EQUATIONS—STOICHIOMETRY—NOMENCLATURE

No experiments. **61 to 66**

CHAPTER IV

EXPERIMENTS ILLUSTRATING THE RELATION BETWEEN VOLUME, PRESSURE, AND TEMPERATURE OF GASES

1. The Law of Boyle

Relation between Volume and Pressure of Gases

66 **Specific problem.**—To investigate the relation between the volume and the pressure of a confined mass of air, its temperature remaining constant.

FIG. 3.—Apparatus to show Boyle's law.

Use a gasometric tube of 25 cubic centimeters in capacity, graduated in fifths (the inside must be dry), filled about one third or one half with air, the rest with mercury, and inserted, the open end down, in a narrow cylinder containing mercury. Hold the tube, using a paper holder to avoid changing the temperature by contact with the hand, in a fixed position; observe the volume occupied by the gas, reading from the top of the mercury column; measure also, the position being unchanged, the length of the mercury column from its top to the free surface of the mercury in the cylinder, using a metric rule or a common one, according to convenience. Now change the position of the tube vertically, read the new volume, and measure the new length of column. In this manner make the observations in five or six different positions of the tube.

Calculations.—It will be found convenient to record the volumes in a vertical column, and the lengths in a column parallel with this, each corresponding horizontally with its proper volume. Deduct the several lengths of mercury column from the length of the mercury column in the barometer, and the differences thus obtained measure the relative pressures of the gas when it occupies the corresponding volumes. Now, looking for a relation between the numbers measuring volume and those measuring pressure, multiply each volume by its corresponding pressure and compare the products. (See Appendix, 15 B.) 66/1

How comes it that pressure is measured in linear units? And why is the observed pressure obtained by subtracting the observed length of column from the barometric length?

What is the limit of accuracy in your measurement of volume? Of pressure? How much variation from constancy may the products show and still prove constancy within the limits of observational accuracy? What does constancy in the products prove concerning the factors?

2. The Law of Charles

Relation between Volume and Temperature of Gases

Specific problem.—To investigate the relation between the volume and the temperature of a confined mass of air, its pressure remaining constant. 67

Use an apparatus of the following description: A gasometric tube of 25 cubic centimeters in capacity, graduated in fifths (the inside must be dry), filled about one third or one half with air, and the rest with mercury. This is inverted, and the open end inserted in a shallow vessel containing mercury. Outside the gasometric tube is a larger glass tube, serving as a jacket, which is closed at the lower end by a stopper, through which pass centrally the gasometric tube and, either side of this, one small tube to carry steam and one to drain off the water. A thermometer is suspended beside the gasometric tube from a stopper which

loosely fits the upper end of the jacket. A flask is connected so that steam may be passed into the jacket at will, and the whole apparatus is fixed firmly in position.

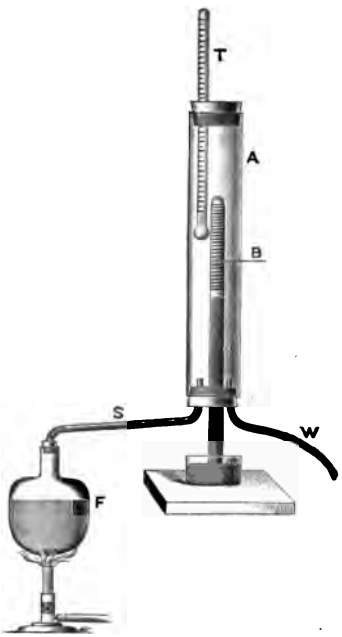

FIG. 4.—Apparatus to show Charles's law. *A*, jacket; *B*, gasometric tube containing air; *T*, thermometer; *S*, rubber tube to convey steam from the flask, *F*; *W*, tube to carry off water.

When it has stood long enough to make sure that the inclosed air has the temperature indicated by the thermometer, read the volume, pressure, and temperature. Then apply heat to the flask containing water, and pass steam into the jacket until the thermometer reaches its maximum and holds it steadily for fifteen or twenty minutes. Finally, read again the volume, pressure, and temperature. Calculate the average increase in volume for 1°, on unit volume, at the initial temperature, pressure being constant. To put this result into conventional form, so that it shall be comparable with the result as given in books, it is only necessary to assume the same rate of change between the initial temperature and 0°, that you have observed between the initial and final temperatures, and from this, to calculate the average increase in volume per degree on unit volume at 0°. This is called *the increment of volume*, also *the coefficient of expansion.*

[What would be the effect of moisture in the gasometric tube on your experimental result?]

CHAPTER V

EXPERIMENTS ILLUSTRATING THE RELATION BETWEEN EQUIVALENT AND COMBINING WEIGHTS AND CERTAIN SPECIFIC PROPERTIES

1. The Law of Gay-Lussac

Relation between Equivalent and Combining Weights of Gases, Elementary and Compound, and their Specific Gravities

Specific problem.—To determine experimentally the specific gravity (A) of oxygen, (B) of carbon dioxide, and to investigate the numerical relation between these values and the combining weights of the substances, assuming that the formula of the latter is CO_2. 71

[What is the specific gravity by definition? (see No. 7, Part I.) What are these combining weights, numerically?]

Assume that the weight of one liter of hydrogen at 0° and 760 millimeters is known to be 0.0899 of a gram. The experimental problem then becomes, to determine the weight of one liter of the gas in question at 0° and 760 millimeters; or, if preferred, to determine the weight of any measured volume of the gas at an observed temperature and pressure. The most direct method of solution would be to weigh a mass of the gas in a vessel of measured capacity; but the experimental difficulties in doing this make it impracticable for beginners. (Can you suggest some of the difficulties?) A less direct method is therefore followed.

A

To determine the weight of one liter of oxygen:

71/A Use a large test-tube (about 230 millimeters or 9 inches long) fitted with a rubber stopper, carrying a glass and rubber delivery tube, like that of the gas generator. The test-tube must be thoroughly dry.

Weigh out roughly about 5 grams of manganese dioxide (symbol MnO_2). Place this in the porcelain crucible and ignite it with the full heat of the flame for ten or fifteen minutes to insure complete dryness.

Weigh out roughly about 20 grams of powdered potassium chlorate (symbol $KClO_3$). Mix the two substances thoroughly in a clean mortar; transfer to the test-tube, and wipe any adhering dust from the upper part of the tube. Insert, to lie just back of the stopper, a loose plug about 15 millimeters (one half inch) wide, made of a coiled strip of asbestos board or of glass wool.

Weigh carefully, to hundredths, the tube and contents thus prepared.

Support the tube horizontally between two rings of the iron stand, and tap it gently so that the powder shall lie somewhat spread out. Insert the stopper and connect with the large collecting bottle in the tank.

Recall the experiment (No. $41/_3$ c) with hydrogen under Law 4, Chapter II, and follow here the details of manipulation there directed for the collection and measurement of the gas generated.

Heat the mixture gently so as to avoid too rapid evolution of gas. It is best to apply heat to only a small portion of the mixture at a time, for, if the whole mass becomes heated, the evolution of gas will not cease when the flame is withdrawn, and more than the bottleful may be generated. When nearly sufficient gas to fill the bottle has been collected, withdraw the heat, and when bubbles cease to pass, remove the stopper, lest water be drawn into the

tube; let the tube cool, and carefully weigh it. The loss in weight is taken as the weight of the oxygen which has passed out of the tube.

The volume of this gas is measured in the collecting bottle, as before indicated. It is well to shake the gas with the water remaining in the bottle, and then to reopen the bottle for a second under water. (Why?)

Calculations.—From this weight and this volume calculate the weight of one liter of oxygen at 0° and 760 millimeters, and from this value calculate the specific gravity of the gas and compare it with its combining weight. 71/1

Make a second determination of the same. The original charge will be more than sufficient to generate two bottlefuls.

Secondary observation.—After the second determination, collect some of the gas in a small bottle and test it with a lighted match. (See Appendix, 19, I and IV.) 71/2

EXPLANATORY NOTE—The potassium chlorate, symbol $KClO_3$, by heating loses its oxygen and becomes potassium chloride, symbol KCl. The temperature at which this decomposition takes place is lowered by the presence of the manganese dioxide, hence its use.

B. First Step

To determine the weight of the carbon dioxide generated from a weighed quantity of calcium carbonate by the action of an acid:

Weigh out with care 5.00 grams of this substance in powder. Transfer it without loss to a filter paper, wiping off any dust adhering to the glass crystal with a small piece of paper. Wrap the whole in a small package, and put it in a beaker. Weigh this beaker with its contents. 81/1

In a second beaker put about 20 grams (15 c. c.) of nitric acid and a glass rod, and weigh this beaker with its contents. Cautiously pour acid from the second beaker into the first, a little at a time, until there is no further action. Then weigh

again each beaker and its contents. The loss of weight in the system is taken as the weight of carbon dioxide generated. Make at least two determinations of this quantity.

B. Second Step

To determine the volume of the gas generated by the action of an acid on a weighed quantity of calcium carbonate:

81/2 Weigh out 10.00 grams of the calcium carbonate, wrap in a paper as in No. $81/_1$, and transfer without loss to the gas generator. Now, carbon dioxide, being soluble in water, can not be collected over water without loss. The difficulty is avoided by using an intermediate bottle, holding about four times as much as the collecting bottle. It is provided with a two-holed stopper, through one hole of which passes a bent glass tube, terminating close to the bottom of the bottle; through the second hole passes a tube terminating just below the stopper, and to the outer end of this is attached a rubber tube carrying the delivery tube. The delivery tube of the generator is now connected with the first tube of the large bottle, and the delivery tube of the latter is inserted in the mouth of the collecting bottle. The gas from the generator will thus be passed to the bottom of the large bottle and, being considerably heavier than air, will lie there some little time, while an equal volume of air will pass out from the top and may be collected and measured, as in the hydrogen experiment. After the connections are made the apparatus should be tested for leakage (see Exp. $41/_3$b).

Pour about 40 grams (30 c. c.) of nitric acid, a little at a time, through the thistle-tube until the reaction ceases. Note the volume of the acid used (why?), the temperature of the water in the tank, that of the room, and the height of the barometer. Make thus at least two determinations of the volume of gas generated.

Calculations.—From these observations calculate first the weight of one liter of the gas at 0° and 760 millimeters, then the specific gravity referred to hydrogen, and finally the ratio of the specific gravity to the combining weight. 81/3

NOTE.—The intermediate bottle, after once serving, before being again used, must be cleaned of its gas. This may be done by blowing it out with a pair of bellows, the nozzle of which, extended by a rubber tube, reaches to the bottom of the bottle. 81/4

EXPLANATORY NOTE.—In this reaction the factors are calcium carbonate (a salt) and nitric acid; the products are calcium nitrate (a soluble salt), water, and carbon dioxide, the latter appearing as a gas. This may be expressed by equation, thus: 81/5

$$\underset{\text{calc. carb.}}{CaCO_3} + \underset{\text{nitric acid.}}{2HNO_3} = \underset{\text{calc. nitrate.}}{Ca(NO_3)_2} + \underset{\text{water.}}{H_2O} + \underset{\text{carbon dioxide.}}{CO_2}$$

Recall the similar reaction in the experiment under Law 1, Chapter II (No. 36).

2. The Law of Dulong and Petit

Relation between Equivalent and Combining Weights of Elementary Solids and their Specific Heats

Definition of specific heat.—The specific heat of a substance is the quantity of heat required to raise in temperature 1 gram of the substance 1°, divided by the quantity of heat required to raise 1 gram of water 1°. 95

It is necessary to discriminate carefully between quantity of heat and temperature. The unit of quantity (see No. $50/_1$) is called the *gram-calorie*, and is the quantity of heat required to raise one gram of water one degree (strictly from 0° to 1°, practically from 19° to 20° or thereabouts). Therefore specific heat may better be defined as the quantity of heat, measured in gram-calories, required to raise one gram of the substance one degree.

95/1 **Specific problem.**—To determine experimentally the specific heat, and to investigate the numerical relation between this value and the combining weight in the case (A) of lead, (B) of zinc, (C) of tin.*

A

95/2 **Specific heat of lead.**—Weigh out carefully 100.0 grams of lead (shot), transfer it to a large dry test-tube, set the tube and contents in water contained in a suitable vessel and boil the water. The lead should be immersed below the surface of the water.

Weigh out carefully in a beaker 50.0 grams of water which has the temperature of the room; test with the thermometer to see that the temperature does not change in an interval of five minutes. Put the thermometer, first drying it, in the lead, and see that it reaches a maximum which it holds for at least five minutes. Record this temperature. Then put the thermome[illegible] k in the water, stir, read the temperature carefully [illegible] rd it.

Now, as quickly as possible, tak[illegible] ube from the water, pour the warm [illegible] into the [illegible] the beaker contains, stir gentl[illegible] e the [illegible] the maximum tem[illegible] d reco[illegible]

The observatio[illegible] re: [illegible] and its temperatu[illegible] mix[illegible] lead and its tempe[illegible] efore[illegible] ture of the resulta[illegible]

95/3 **Calculations.**—L[illegible]

x = the quantity of [illegible] to [illegible]
y = the quantity of [illegible]
t = the degrees of [illegible]
t' = the degrees of t[illegible]

* One of these three [illegible] perhaps all may be omi[illegible] ones in physics.

It follows that

$100 \times t \times x =$ the quantity of heat lost by 100 grams of lead in cooling $t°$.

$50 \times t' \times y =$ the quantity of heat gained by 50 grams of water in warming $t'°$.

If now the experimental conditions are made such that the lead shall lose no heat save what goes to the water, and the water gain no heat save what comes from the lead, then these two quantities of heat must be equal. Equating the two expressions gives

$$100 \times t \times x = 50 \times t' \times y.$$

From which is obtained

$$\frac{x}{y} = \frac{50 \times t'}{100 \times t} = \text{Specific heat by definition.}$$

The experimental conditions called for to justify this equation can be but crudely realized by the directions here given, but sufficiently to give a good illustration of method. Calculate the quantity of heat required to raise 205 grams of lead 1°.

B

ecific heat of zinc.—Weigh out carefully 50.0 grams of 95/4
ed zinc, and 50.0 grams of water. The procedure
lations are the same as in the case of lead. Cal-
uantity of heat required to raise 65 grams of

C

of tin.—Use 50.0 grams of tin in coarse 95/5
grams of water, and proceed as in B.
ity of heat required to raise 118 grams

l used in these experiments, so that it may
e again.

95/1 **Specific problem.**—To determine experimentally the specific heat, and to investigate the numerical relation between this value and the combining weight in the case (A) of lead, (B) of zinc, (C) of tin.*

A

95/2 **Specific heat of lead.**—Weigh out carefully 100.0 grams of lead (shot), transfer it to a large dry test-tube, set the tube and contents in water contained in a suitable vessel, and boil the water. The lead should be immersed below the surface of the water.

Weigh out carefully in a beaker 50.0 grams of water which has the temperature of the room; test with the thermometer to see that the temperature does not change in an interval of five minutes. Put the thermometer, first drying it, in the lead, and see that it reaches a maximum which it holds for at least five minutes. Record this temperature. Then put the thermometer back in the water, stir, read the temperature carefully, and record it.

Now, as quickly as possible, take the test-tube from the water, pour the warm lead into the water which the beaker contains, stir gently with the thermometer, quickly read the maximum temperature and record it.

The observations therefore are: The weight of the water and its temperature just before mixing; the weight of the lead and its temperature just before mixing; the temperature of the resultant mixture.

95/3 **Calculations.**—Let

x = the quantity of heat required to raise 1 gram of lead 1°;
y = the quantity of heat required to raise 1 gram of water 1°;
t = the degrees of temperature lost by the lead;
t' = the degrees of temperature gained by the water.

* One of these three determinations may be thought sufficient, or perhaps all may be omitted if the student has already made similar ones in physics.

It follows that

$100 \times t \times x =$ the quantity of heat lost by 100 grams of lead in cooling $t°$.

$50 \times t' \times y =$ the quantity of heat gained by 50 grams of water in warming $t'°$.

If now the experimental conditions are made such that the lead shall lose no heat save what goes to the water, and the water gain no heat save what comes from the lead, then these two quantities of heat must be equal. Equating the two expressions gives

$$100 \times t \times x = 50 \times t' \times y.$$

From which is obtained

$$\frac{x}{y} = \frac{50 \times t'}{100 \times t} = \text{Specific heat by definition.}$$

The experimental conditions called for to justify this equation can be but crudely realized by the directions here given, but sufficiently to give a good illustration of method. Calculate the quantity of heat required to raise 205 grams of lead 1°.

B

Specific heat of zinc.—Weigh out carefully 50.0 grams of granulated zinc, and 50.0 grams of water. The procedure and calculations are the same as in the case of lead. Calculate the quantity of heat required to raise 65 grams of zinc 1°. 95/4

C

Specific heat of tin.—Use 50.0 grams of tin in coarse powder, and 50.0 grams of water, and proceed as in B. Calculate the quantity of heat required to raise 118 grams of tin 1°. 95/5

NOTE.—Save the material used in these experiments, so that it may be dried and be ready for use again.

95/1 **Specific problem.**—To determine experimentally the specific heat, and to investigate the numerical relation between this value and the combining weight in the case (A) of lead, (B) of zinc, (C) of tin.*

A

95/2 **Specific heat of lead.**—Weigh out carefully 100.0 grams of lead (shot), transfer it to a large dry test-tube, set the tube and contents in water contained in a suitable vessel, and boil the water. The lead should be immersed below the surface of the water.

Weigh out carefully in a beaker 50.0 grams of water which has the temperature of the room; test with the thermometer to see that the temperature does not change in an interval of five minutes. Put the thermometer, first drying it, in the lead, and see that it reaches a maximum which it holds for at least five minutes. Record this temperature. Then put the thermometer back in the water, stir, read the temperature carefully, and record it.

Now, as quickly as possible, take the test-tube from the water, pour the warm lead into the water which the beaker contains, stir gently with the thermometer, quickly read the maximum temperature and record it.

The observations therefore are: The weight of the water and its temperature just before mixing; the weight of the lead and its temperature just before mixing; the temperature of the resultant mixture.

95/3 **Calculations.**—Let

x = the quantity of heat required to raise 1 gram of lead 1°;
y = the quantity of heat required to raise 1 gram of water 1°;
t = the degrees of temperature lost by the lead;
t' = the degrees of temperature gained by the water.

* One of these three determinations may be thought sufficient, or perhaps all may be omitted if the student has already made similar ones in physics.

It follows that

$100 \times t \times x =$ the quantity of heat lost by 100 grams of lead in cooling $t°$.

$50 \times t' \times y =$ the quantity of heat gained by 50 grams of water in warming $t'°$.

If now the experimental conditions are made such that the lead shall lose no heat save what goes to the water, and the water gain no heat save what comes from the lead, then these two quantities of heat must be equal. Equating the two expressions gives

$$100 \times t \times x = 50 \times t' \times y.$$

From which is obtained

$$\frac{x}{y} = \frac{50 \times t'}{100 \times t} = \text{Specific heat by definition.}$$

The experimental conditions called for to justify this equation can be but crudely realized by the directions here given, but sufficiently to give a good illustration of method. Calculate the quantity of heat required to raise 205 grams of lead 1°.

B

Specific heat of zinc.—Weigh out carefully 50.0 grams of granulated zinc, and 50.0 grams of water. The procedure and calculations are the same as in the case of lead. Calculate the quantity of heat required to raise 65 grams of zinc 1°. 95/4

C

Specific heat of tin.—Use 50.0 grams of tin in coarse powder, and 50.0 grams of water, and proceed as in B. Calculate the quantity of heat required to raise 118 grams of tin 1°. 95/5

NOTE.—Save the material used in these experiments, so that it may be dried and be ready for use again.

95/1 **Specific problem.**—To determine experimentally the specific heat, and to investigate the numerical relation between this value and the combining weight in the case (A) of lead, (B) of zinc, (C) of tin.*

A

95/2 **Specific heat of lead.**—Weigh out carefully 100.0 grams of lead (shot), transfer it to a large dry test-tube, set the tube and contents in water contained in a suitable vessel, and boil the water. The lead should be immersed below the surface of the water.

Weigh out carefully in a beaker 50.0 grams of water which has the temperature of the room; test with the thermometer to see that the temperature does not change in an interval of five minutes. Put the thermometer, first drying it, in the lead, and see that it reaches a maximum which it holds for at least five minutes. Record this temperature. Then put the thermometer back in the water, stir, read the temperature carefully, and record it.

Now, as quickly as possible, take the test-tube from the water, pour the warm lead into the water which the beaker contains, stir gently with the thermometer, quickly read the maximum temperature and record it.

The observations therefore are: The weight of the water and its temperature just before mixing; the weight of the lead and its temperature just before mixing; the temperature of the resultant mixture.

95/3 **Calculations.**—Let

x = the quantity of heat required to raise 1 gram of lead 1°;
y = the quantity of heat required to raise 1 gram of water 1°;
t = the degrees of temperature lost by the lead;
t' = the degrees of temperature gained by the water.

* One of these three determinations may be thought sufficient, or perhaps all may be omitted if the student has already made similar ones in physics.

It follows that

$100 \times t \times x =$ the quantity of heat lost by 100 grams of lead in cooling $t°$.

$50 \times t' \times y =$ the quantity of heat gained by 50 grams of water in warming $t'°$.

If now the experimental conditions are made such that the lead shall lose no heat save what goes to the water, and the water gain no heat save what comes from the lead, then these two quantities of heat must be equal. Equating the two expressions gives

$$100 \times t \times x = 50 \times t' \times y.$$

From which is obtained

$$\frac{x}{y} = \frac{50 \times t'}{100 \times t} = \text{Specific heat by definition.}$$

The experimental conditions called for to justify this equation can be but crudely realized by the directions here given, but sufficiently to give a good illustration of method. Calculate the quantity of heat required to raise 205 grams of lead 1°.

B

Specific heat of zinc.—Weigh out carefully 50.0 grams of granulated zinc, and 50.0 grams of water. The procedure and calculations are the same as in the case of lead. Calculate the quantity of heat required to raise 65 grams of zinc 1°. 95/4

C

Specific heat of tin.—Use 50.0 grams of tin in coarse powder, and 50.0 grams of water, and proceed as in B. Calculate the quantity of heat required to raise 118 grams of tin 1°. 95/5

NOTE.—Save the material used in these experiments, so that it may be dried and be ready for use again.

95/1 **Specific problem.**—To determine experimentally the specific heat, and to investigate the numerical relation between this value and the combining weight in the case (A) of lead, (B) of zinc, (C) of tin.*

A

95/2 **Specific heat of lead.**—Weigh out carefully 100.0 grams of lead (shot), transfer it to a large dry test-tube, set the tube and contents in water contained in a suitable vessel, and boil the water. The lead should be immersed below the surface of the water.

Weigh out carefully in a beaker 50.0 grams of water which has the temperature of the room; test with the thermometer to see that the temperature does not change in an interval of five minutes. Put the thermometer, first drying it, in the lead, and see that it reaches a maximum which it holds for at least five minutes. Record this temperature. Then put the thermometer back in the water, stir, read the temperature carefully, and record it.

Now, as quickly as possible, take the test-tube from the water, pour the warm lead into the water which the beaker contains, stir gently with the thermometer, quickly read the maximum temperature and record it.

The observations therefore are: The weight of the water and its temperature just before mixing; the weight of the lead and its temperature just before mixing; the temperature of the resultant mixture.

95/3 **Calculations.**—Let

$x =$ the quantity of heat required to raise 1 gram of lead 1°;
$y =$ the quantity of heat required to raise 1 gram of water 1°;
$t =$ the degrees of temperature lost by the lead;
$t' =$ the degrees of temperature gained by the water.

* One of these three determinations may be thought sufficient, or perhaps all may be omitted if the student has already made similar ones in physics.

It follows that

$100 \times t \times x =$ the quantity of heat lost by 100 grams of lead in cooling $t°$.

$50 \times t' \times y =$ the quantity of heat gained by 50 grams of water in warming $t'°$.

If now the experimental conditions are made such that the lead shall lose no heat save what goes to the water, and the water gain no heat save what comes from the lead, then these two quantities of heat must be equal. Equating the two expressions gives

$$100 \times t \times x = 50 \times t' \times y.$$

From which is obtained

$$\frac{x}{y} = \frac{50 \times t'}{100 \times t} = \text{Specific heat by definition.}$$

The experimental conditions called for to justify this equation can be but crudely realized by the directions here given, but sufficiently to give a good illustration of method. Calculate the quantity of heat required to raise 205 grams of lead 1°.

B

Specific heat of zinc.—Weigh out carefully 50.0 grams of granulated zinc, and 50.0 grams of water. The procedure and calculations are the same as in the case of lead. Calculate the quantity of heat required to raise 65 grams of zinc 1°. 95/4

C

Specific heat of tin.—Use 50.0 grams of tin in coarse powder, and 50.0 grams of water, and proceed as in B. Calculate the quantity of heat required to raise 118 grams of tin 1°. 95/5

NOTE.—Save the material used in these experiments, so that it may be dried and be ready for use again.

3. The Law of Mitscherlich

Relation between Composition, and hence Combining Weight, and Specific, i. e., Crystalline Form

107 No experiments.

4. The Law of Raoult (I)

Relation between Combining Weights of Solutes and Specific Depressions of the Freezing Point in Specified Solvent (See Part I, Nos. 21 and 23/1)

111 **Specific problem.**—I. To investigate experimentally the relation between the depression of the freezing point and the quantity of the solute (A) when the solute is camphor and the solvent paraffin; and (B) when the solute is naphthalene and the solvent paraffin.

II. To investigate the numerical relation between the specific depression (i. e., depression for 1 gram of solute in 100 grams of solvent) of camphor in paraffin, and of naphthalene in paraffin, and the combining weights of the solutes.

A

111/1 (1) Weigh out carefully 10.00 grams of paraffin and 0.80 of a gram of camphor. Melt the paraffin in an evaporating dish on the water-bath, then add the camphor and let it dissolve, stirring well. Pour a suitable portion of the solution into a clean, dry test-tube. Let the liquid be about one inch deep. In another tube put some paraffin. Have a stirring-rod in each tube, and attach the tubes by rubber bands to a thermometer. Suspend the tubes in a beaker containing water, so that they are suitably immersed, and apply heat until the mixture melts. Allow to cool very slowly, keeping the mixtures and the water well stirred, and observe the freezing points. Repeat several times for constant results. It will save time to prepare the solution

called for in Exp. 111/2, and to attach the three tubes at one time to the thermometer.

(2) Make a mixture of 10.00 grams of paraffin and 1.20 grams of camphor, as in (1), and in a similar manner determine its freezing point. Are the depressions approximately proportional to the quantities of the solute in a constant quantity of the solvent? 111/2

Calculate the specific depressions—i. e., depressions for 1 gram of solute in 100 grams of solvent—and take their average.

B

(3) Make a mixture of 10.00 grams of paraffin and 0.60 of a gram of naphthalene, as in A; (4) also of 10.00 grams of paraffin and 1.20 grams of naphthalene. Observe the freezing points, and make the calculations as before. 111/3 111/4

II. Multiply the specific depressions of camphor in paraffin by the combining weight of camphor ($C_{10}H_{16}O$ = 151), and the specific depression of naphthalene by the combining weight of naphthalene ($C_{10}H_8$ = 127), and compare the products. Are they approximately constant? 111/5

NOTE.—It will be realized that the probable error of reading on the ordinary thermometer is so large a fraction of the depression itself, that the results can not be quantitatively very satisfactory. A thermometer reading at least to hundredths would be needed for good results. Calculate the variation in the product made by the variation of one quarter of a degree in the depression. 111/6

NOTE.—The tubes may be cleaned by immersing in boiling water, and the dishes by wiping with filter paper while still warm. 111/7

5. The Law of Raoult (II)

Relation between Combining Weights of Solutes and Specific Elevations of Boiling Temperature in Specified Solvent
(See Part I, Nos. 21 and 24/4)

Specific problem.—I. To investigate experimentally the relation between the elevation of the boiling temperature 127

and the quantity of the solute (A) when the solute is sodium acetate and the solvent water, and (B) when the solute is potassium tartrate and the solvent water.

II. To investigate the numerical relation between the specific elevation of boiling temperature—i. e., elevation for 1 gram of solute in 100 grams of solvent—by sodium acetate in water, and by potassium tartrate in water, and the combining weights of the solutes.

A

127/1 (1) Use a small distilling flask, as in the observation of boiling point (see Appendix, 17). Put in it a suitable quantity of water and some bits of pumice (why the latter? See No. $24/_2$, Part I), boil, and observe carefully the temperature of the boiling water. Repeat all readings for constant results.

127/2 (2) Drain the water thoroughly from the flask, and put in its place a solution of 100.0 grams (or 100 cubic centimeters) of water and 15.00 grams of sodium acetate with clean pumice. Again observe the temperature of the boiling solution.

127/3 (3) In a similar manner observe the boiling temperature of a solution containing 100.0 grams (or 100 cubic centimeters) of water and 30.00 grams of sodium acetate.

Are the elevations approximately proportional to the quantities of the solute in a constant quantity of the solvent? Calculate the specific elevations, and take their average.

B

127/4 (4) Observe the boiling temperature of a solution of 100.0 grams (or 100 cubic centimeters) of water and 20.00
127/5 grams of potassium tartrate; (5) also of 100.0 grams (or 100 cubic centimeters) of water and 40.00 grams of potassium tartrate. Make the same calculations as before.

II. Multiply the specific elevation of sodium acetate 127/6
($NaC_2H_3O_2$) in water by the combining weight of the same, and the specific elevation of potassium tartrate ($K_2C_4H_4O_6$) by its combining weight, and compare the products. Are they approximately constant?

The same remark as to accuracy of observation applies here as under the preceding law. Care should be taken that the bulb of the thermometer is completely immersed in the liquid.

NOTE.—The solutions may be saved in order to recover the salts by crystallization.

Alternative experiment.—Instead of the solutions indi- 127/7
cated in the preceding experiment, may be used the following: A 15.40 per cent and a 30.80 per cent solution of potassium chloride (KCl), and an 11.00 per cent and a 22.00 per cent solution of ammonium chloride (NH_4Cl).

CHAPTER VI

EXPERIMENTS ILLUSTRATING THE METHOD OF DETERMINING EQUIVALENT AND COMBINING WEIGHTS OF ELEMENTS AND FORMULAS OF COMPOUNDS

1. Determination of the Equivalent Weight of an Element

144 **Specific problem.**—To determine experimentally the mass of tin which combines with 7.94 grams of oxygen. (Tin does not combine with hydrogen.)

144/1 EXPLANATORY NOTE.—It is not practicable to cause a weighed quantity of tin to combine with oxygen of the air, and to weigh the oxide obtained; but by the action of nitric acid, the oxidizing power of which has already been noted [in what connection?], an oxide of tin is produced which does not volatilize, does not combine with nitric acid, and does combine with water, forming a compound, however, from which the water is expelled by prolonged heating, leaving simply the oxide. The excess of nitric acid is also volatilized by heat.

144 Weigh very carefully to hundredths of a gram a small evaporating dish, clean and dry, together with a short glass rod.

Weigh out carefully 5.00 grams of pure tin foil. Put a portion of this in small pieces in the evaporating dish, and just moisten with nitric acid. When the action is nearly over, put in more tin, and then moisten again with acid. Continue this until all the tin is used, with the minimum of acid that will suffice.

Describe what takes place. What is the brown gas produced? Have care not to inhale it; it is well to use the hood. Also bear in mind the extreme corrosiveness of nitric acid. Use the rod to pour by, leaving it in the dish. Keep the outside of the beaker clean. Have a wet sponge at hand.

Heat the dish and contents on the water-bath until the liquid has entirely volatilized, transfer to the iron ring of the stand, apply the direct flame, gently at first passing it back and forth, then give it the full heat of the burner, continuing for some time after the fumes have ceased to appear. The powder should be slightly brown when hot, and yellowish when cool.

Let the dish become thoroughly cool, and weigh it with its contents to hundredths of a gram. To insure the completeness of the operation, ignite again for fifteen or twenty minutes, let cool, and weigh again.

Assuming that you have in the dish a compound containing only tin and oxygen, calculate the weight of oxygen which has combined with the 5 grams of tin, and from this the weight of tin which combines with 7.94 grams of oxygen. Make at least two complete determinations. 144/2

2. Determination of the Combining Weight of an Element

Under the law of Dulong and Petit choose what multiple of the equivalent weight determined in No. 144/2 shall be taken as the combining weight of tin. The specific heat of tin is 0.056. (See Nos. 101 and 105, Part I.) 150

It will be realized that the utmost care must be taken in the manipulation, since the weighings must be to hundredths of a gram, and the variation in the quantity of oxygen for 5 grams of tin should not be more than one or two hundredths of a gram. 150/1

Having determined the combining weight of tin, deduce the formula for the oxide. Try to write the equation for the reaction between tin (symbol, Sn) and nitric acid, HNO_3. What is the percentage of tin and of oxygen in tin oxide? 150/2

3. Determination of the Formula of a Compound

Specific problem.—To determine the percentage of the proximate constituents, carbon dioxide, CO_2, and sodium 154

oxide, Na_2O, in the compound, sodium carbonate, and from these to deduce the formula of the compound.

154/a (*a*) To determine the percentage of carbon dioxide: Take a sufficient quantity of the sodium carbonate for all the following experiments, heat in the larger evaporating dish over the flame, gently and not too hot. This is simply to secure the dryness of the sample.

Weigh out carefully 5.00 grams of the dry sample, and follow the procedure given for determining the weight of carbon dioxide in calcium carbonate (see Exp. $81/_1$), using hydrochloric instead of nitric acid. Make at least two determinations.

Calculate the result as grams of carbon dioxide in 100 grams of the sample.

Secondary observation.—Filter and crystallize the salt which is left in solution. What substance is it?

(*b*) To determine the percentage of sodium oxide:

154/1 EXPLANATORY NOTE.—Since it is not practicable to separate this constituent from the compound, and weigh it by itself, it is necessary to convert it quantitatively into some substance which it is practicable to separate and weigh, and the composition of which is known. For this purpose the carbonate is converted into sodium nitrate by the action of nitric acid, the carbon dioxide escapes, and the water and excess of nitric acid are volatilized. Sodium nitrate is not volatile, and is not decomposed by heating short of fusion. Its composition is given by the formula $Na_2N_2O_6$, or, as usually expressed, $NaNO_3$. [What percentage of Na_2O does this formula show?]

154/b Weigh out carefully 5.00 grams of the dry sample of sodium carbonate in a small evaporating dish, the weight of which has been determined to hundredths of a gram. Neutralize this with nitric acid, using a very slight excess, and having care not to lose by effervescence. Evaporate on the water-bath until the liquid has disappeared, and then, passing the burner back and forth, heat gently until the substance just begins to melt. Let cool, and weigh. The substance obtained is sodium nitrate. Make thus at

least two determinations of the weight of this substance obtained from 5 grams of the carbonate.

Knowing the percentage of sodium oxide in sodium nitrate, calculate the weight of sodium oxide contained in the weight of the nitrate obtained, and reckon this result as grams of the oxide in 100 grams of the carbonate.

Secondary observation.—Redissolve and crystallize the sodium nitrate.

(*c*) To deduce the formula:

Since the proximate constituents are carbon dioxide, 155
CO_2, and sodium oxide, Na_2O, the formula must be $(Na_2O)_x(CO_2)_y$, the coefficients, x and y, to be deduced from the percentages experimentally obtained. Hence divide the percentage of each constituent by the combining weight of the same, and the quotients give the ratio of the coefficients, x and y. Now these quotients are not necessarily whole numbers, but the peculiarity of chemical phenomena is that they stand in the ratio of whole numbers, usually quite small. Hence inspection or division by the smallest will suffice to show the smallest whole numbers that have the ratio of the quotients. In this specific problem the approach of the quotients to equality measures the accuracy of the experimental work. The simplest values, therefore, for x and y are 1 and 1, and, in the properties of this substance, no reason is found for using any multiples of these values. The accepted formula is therefore $Na_2O{\cdot}CO_2$, or, as it may also be written, Na_2CO_3.

[Write the equation for the reaction between sodium carbonate and nitric acid.]

CHAPTER VII

THE ATOMIC THEORY

157 to 187 No experiments.

CHAPTER VIII

RELATION BETWEEN THE PROPERTIES OF THE ELEMENTS IN GENERAL AND THEIR COMBINING WEIGHTS

Experimental Study of the Properties of the First Twenty-five Elements (in the Order of their Combining Weights) and Some of their Compounds

So far as practicable, each element in turn will be presented in its free condition for descriptive study: first as to its physical properties, those which appear on inspection, and others; second, as to its chemical properties. Then will follow the study of some of its most important compounds. It is judged unnecessary to give detailed directions for manipulation in all experiments, as by this time the student should have had sufficient experience in the laboratory to give him some judgment of his own as to how to do things. Some of the manipulations, too, are repeated many times.

1. HYDROGEN

Symbol H.—Comb. wt. 1

202 **Preparation.**—See Exps. $17/_1$ and $41/_4$ and Appendix, 19, I. Use the small collecting bottles or test-tubes. Use iron (nails) or zinc and hydrochloric or dilute sulphuric acid.

NOTE.—*Hydrogen with air makes a mixture which may explode dangerously on ignition. Therefore the greatest*

caution must always be taken to avoid accident from unexpected ignition by contact with flame.

Physical Properties

Observe as to *color*, *odor* (any odor is due to impurity). 203

As to weight compared with air.—A crude test: Fill a test-tube with hydrogen over water, close with the thumb, hold its mouth downward directly over and close to the mouth of a second tube, then remove the thumb quickly, bringing the two ends together, reverse the position of the tubes, separate them, closing the second, now the upper one, with the thumb, and test at the flame for the presence of hydrogen in both tubes.

As to solubility in water.—A crude test: Collect in a 204
test-tube, leaving a little water; close tightly with the thumb, shake, open under water, close, and shake again. Repeat this several times, and note if the volume of water in the tube increases and the gas decreases.

As to relative diffusibility.—Use a dry glass tube, closed 203/1
at one end with a porous plug of plaster of Paris, and open at the other. Close temporarily the plugged end with the thumb or a cork, and fill the tube thoroughly by dry displacement—i. e., hold the tube open end downward, thrust the delivery tube at first well up to the top, then slowly withdraw it (compare Appendix, 19, VI). When the tube is full, immerse the lower, open end in water, uncover the porous plug, and allow to stand for some time. What takes place? Can you explain the phenomenon?

A modification of the preceding experiment, suitable to be performed 203/2
by the teacher before the class: Use a porous battery jar, closed by a cork or a plaster plug through which passes a glass tube of convenient length. Support this so that the lower open end of the tube is immersed in a colored liquid (e. g., water, colored by potassium permanganate). Slowly lower over the porous jar a glass bell jar filled with hydrogen. When the gas ceases to bubble from the end of the tube, remove the bell jar. The same may be tried with illuminating gas in place of hydrogen.

205 **Occlusion.**—Hold a piece of platinum sponge (i. e., finely divided platinum deposited on the surface of asbestos fiber), thoroughly dried, in the stream of hydrogen issuing from the delivery tube, but only when the gas is sufficiently pure to be ignited with safety.

[This experiment, like the preceding one, may best be performed by the teacher.]

Chemical Properties

Test as to the action of hydrogen on wet litmus paper.

207 **Test as to combustibility.**—With suitable precaution, the gas issuing from the delivery tube may be ignited as a jet, but, owing to the danger of explosion, this should not be tried until the action has continued long enough to expel the air from the generator. It is best always to test a small sample of the gas thus: Collect in a test-tube over water, close with the thumb, bring it to the flame, mouth downward, remove the thumb, ignite the gas, and slowly invert the tube. The gas should burn quietly in the tube. If it burns quickly with a slight report, it is not safe to ignite at the generator.

207/1 *Make it a rule always to wrap several folds of the towel over and around the generator before igniting the gas, no matter how confident you may be of its purity. Serious accident may follow neglect of this precaution.*

Observe the character of the hydrogen flame; hold a dry glass plate or beaker over the flame. What is the product of combustion?

208 **Nascent state.**—Use four test-tubes, fill about two thirds full with water, and color each portion slightly with a few drops of potassium permanganate solution. Into the first put a few fragments of zinc and a few drops of sulphuric acid, and let the action continue for some minutes. What is the result? What substance is it which causes the color to disappear? To answer this, place in the second tube some zinc, in the third some sulphuric acid, and into the fourth let the hydrogen from the generator slowly bubble, preferably using zinc as the metal.

EXPLANATORY NOTE.—The permanganate loses its color in consequence of losing some of its oxygen. The oxygen is taken from it by the hydrogen, but only at the moment of its liberation from the acid, when it is said to be in the *nascent state*. The experiment may not be entirely satisfactory, for the reason that the impurity which is usually carried by the hydrogen from the generator, and which comes from the metal, and imparts the odor to the gas, tends to remove oxygen from the permanganate. Observe as to the odor of the gas which has bubbled through the colored liquid. 208/1

To explain the phenomenon of the nascent state it has been suggested that the substance in the atomic condition may show an activity which is wholly or partly lost after the atoms have come together into molecules. 208/2

Another method of generating hydrogen.—Use a few fragments of zinc in a test-tube with a dilute solution of sodium hydroxide. The reaction is facilitated by a piece of iron (a nail) in contact with the zinc. 202/3

2. LITHIUM

Li.—6.97

No experiments, unless to show a sample of some salt of lithium—e. g., the chloride—and the color it imparts to the flame. 210

3. GLUCINUM or BERYLLIUM

Gl. or Be.—9.0

No experiments. 216

4. BORON

B.—10.86

Boron is difficult of preparation, so it is impracticable to show it. It forms an oxide whose symbol is B_2O_3, and which combines with water, forming boric acid. The sodium salt of this is the familiar commercial substance, borax.

Borax and boric acid crystallized.—Saturate about a beakerful of water with borax. Filter, if not clear, and 225

set aside about one half of this to crystallize. To the remainder add about one half its volume of hydrochloric acid, and set this aside to crystallize. Observe the different appearance of the two substances when crystallized.

Rinse a sample of each substance quickly with a very little cold water, and dry it on filter paper. Observe the reactions on litmus paper.

226 **Flame coloration.**—Put a very small quantity of borax in an evaporating dish, add a few drops of alcohol, ignite the alcohol, and observe the color of the flame, while stirring the mixture. Extinguish the flame, add a few drops of dilute sulphuric acid, reignite the alcohol, and again observe the flame color. This test is often used to recognize boric acid and its compounds.

228 **The borax bead.**—Melted borax has the property of dissolving many metallic oxides, and these often impart characteristic colors to the substance. This test is very useful in qualitative analysis. To make a borax bead, use a piece of platinum wire, about 75 millimeters (3 inches) long, one end of which is fused into a piece of glass tubing for a handle. Bend the free end of the wire around the sharpened end of a lead pencil, so as to make a small loop. Heat this red hot, touch it to some powdered borax, and heat the borax which adheres to the wire. What is the first effect of this, and to what is it due? (see Exp. $21/_7$.) When the borax has fused to a clear, glasslike bead, touch it, while still hot, to some particles of copper oxide. These adhere to the hot bead. Do not let it take up too much—a very minute quantity suffices; and if you have not enough on first trial, it is easy to add more, but it is not so easy to reduce the quantity. Heat again to fuse the bead with the adhering copper oxide. The particles of the latter can be seen slowly to dissolve, often with beautiful play of colors on the surface of the bead. Observe the color of the bead, when hot, and when cold. The bead should be transparent, but, if too much material has been put in, it

will be opaque, so the color can not be distinguished. In this event, clean the wire, leaving only a fragment of the bead, and add more borax. To clean the wire, heat the 229
bead hot, and quickly cool it in water. This makes the mixture brittle, so that it can be easily removed. To get the best results, in the bead experiments, the mouth blowpipe should be used for heating. By this means the character of the flame can be so varied as to cause the oxidation of the dissolved substance, or the removal of oxygen from it—i. e., reduction. These changes often produce changes in color. Thus the copper bead is blue when heated in the oxidizing flame, and it has the color of metallic copper in the reducing flame. (Concerning the use of the blowpipe see Appendix 21.)

Manganese in the bead.—After seeing the color of the 228/2
copper bead in the oxidizing and in the reducing flame, make a new bead and color it with manganese dioxide. Observe the color in each flame.

5. CARBON

C.—11.91

Elementary carbon exists in three allotropic forms, 234
diamond, graphite, and charcoal; only the last is here studied.

Preparation.—Heat a small quantity of sugar on the 248
spatula blade. It burns and chars. The black charred material consists mainly of carbon. In similar manner many other substances show the charring effect of heat, and this is evidence of the presence of carbon as a constituent in the substance. It is due to the removal in part of other constituents, particularly hydrogen, by combustion, the less easily combustible carbon being left as residue.

Place some fine shavings in a dry test-tube, fitted with 254
a cork and a short glass delivery tube, and apply the full heat of the flame. Note the appearance of moisture and

white fumes. Ignite the gas issuing from the tube. After the heating observe the black residue in the tube. This is charcoal. Note also the liquid, having tarry odor. Test it with litmus.

The application of heat in this manner, without free access of oxygen or air, is called *dry* or *destructive distillation.* It is applied on a large scale to the production of various substances from wood, and likewise to the production of illuminating gas and other substances by the de-
253 structive distillation of coal. Heat a little soft coal in a test-tube, as in No. 254.

Properties of Carbon (Charcoal)

254/1 **Porosity.**—Holding a piece of charcoal in the tongs, plunge it beneath the surface of some hot water. Is charcoal heavier or lighter than water? Keep it under hot water for a considerable time. Does it finally sink in the water?

255 **Absorptive power.**—Generate a very little hydrogen sulphide, using a piece of iron sulphide not larger than a bean and a few drops of hydrochloric acid, and collecting the gas in a dry bottle covered with a glass plate (Appendix, 19, V). Drop into this a piece of charcoal, previously heated for a few seconds in the gas flame. Set this aside with the cover on, and some time later observe by odor if the gas has been absorbed by the charcoal.

255/1 In a beaker containing water, colored by a few drops of potassium permanganate solution, place a small quantity of boneblack (animal charcoal), boil for a few minutes, then filter. Does the filtrate lose its color? Is the color removed by filtering simply, without the use of the charcoal?

257 **Combustion.**—Does the substance burn? Does it burn with a flame? Why not?

257/1 **Product of combustion.**—Ignite a piece of charcoal in the flame until it is well aglow, then drop it into a bottle and cover the latter with a glass plate. When the coal is ex-

tinguished, either take out the remaining charcoal or invert the bottle over another bottle, pouring thus the heavy gas from the first into the second bottle and leaving the charcoal. Next pour into the second bottle some lime-water (calcium hydroxide solution, CaO_2H_2) and shake the contents. The white precipitate is calcium carbonate, $CaCO_3$, and its formation may generally be taken as evidence of carbon dioxide, which is, in fact, the combustion product of carbon. Write the equation for the formation thus of calcium carbonate.

Reducing power.—Make a shallow hole in a piece of charcoal, place in this a small quantity of litharge (lead oxide), and turn the flame (reducing) of the blowpipe upon this (see Appendix, 21, II). Describe and explain what takes place. 257/2

5a. *Carbon Dioxide, CO_2*

Preparation.—Use calcium carbonate and hydrochloric acid in the generator. Collect the gas by upward displacement of air (Appendix, 19, V) in the small collecting bottles. 261

As to solubility.—Test as in Exp. 204. 262

As to combustion and specific gravity.—Plunge the lighted taper slowly into a bottle filled with the gas. 263

Place the lighted taper in one bottle, and pour upon it the heavy gas from a second bottle. Holding a piece of magnesium ribbon in the tongs, ignite it at the flame and plunge it into a bottle well filled with the gas. What is the white powder seen in the bottle after this experiment? What is the black substance? Why does it retain somewhat the form of the ribbon?

As to acid reaction.—Test the action on wet blue litmus paper. 264

As to reaction with lime-water (calcium hydroxide solution).—Collect some of the gas in a test-tube, pour in some lime-water, and shake. Pass in repeatedly more of the gas 265

until the precipitate at first formed nearly or quite disappears. Then boil the contents of the tube. Explain the reprecipitation.

265/1 EXPLANATORY NOTE.—Excess of carbon dioxide in water forms with calcium carbonate an acid carbonate which is soluble in water. This compound is broken up at the temperature of boiling and the excess of carbon dioxide is expelled, in consequence of which the insoluble calcium carbonate reappears. Many natural waters contain this acid carbonate of calcium or of other bases, and therefore become turbid and deposit sediment by boiling. Test some sample of natural water for this effect. See also Part I, No. 271.

267 **Carbon dioxide in the breath.**—Through a glass tube blow air from the lungs into some lime-water.

5d. *The Hydrocarbons*

Methane, CH_4

276 **Preparation.**—Take about equal bulks of lime, CaO, and of sodium acetate, $NaC_2H_3O_2$; pulverize and mix them thoroughly in the mortar; put some of the mixture in a dry test-tube (about three quarters full), which is provided with a cork and delivery tube; support this on the iron ring, heat, and collect the gas over water in test-tubes.

277 **Properties.**—Examine the gas as to color, odor, solubility in water, and action on litmus (after the sample of gas has been shaken with water). Test for combustibility by igniting a test-tubeful at the flame; then test for a product of combustion by pouring some lime-water into the tube in which the gas has quietly burned, and shaking. What are the products of combustion? The combustibility may be tested also by igniting the gas at the end of the delivery tube.

Acetylene, C_2H_2

280 **Preparation.**—Put three or four lumps of calcium carbide, CaC_2, in the gas generator, with enough alcohol to cover the end of the thistle-tube. Add water through the

thistle-tube, a few drops at a time, so as to regulate the flow of gas, and collect the gas over water in test-tubes as for methane.

Properties.—Examine as to color, odor, solubility in 281
water, and action on litmus, as with methane. Test as to combustibility by igniting a test-tubeful of the gas at the flame, then mix air with acetylene in a test-tube and ignite. Do *not* try to ignite the gas at the delivery tube without the precautions taken with hydrogen (testing a small sample and throwing the towel over the generator), for *a mixture of acetylene and air may be very explosive.*

Describe what takes place in the generator, also the appearance of 281/a
calcium carbide. Is the latter combustible? Allow a lump of it to lie exposed to the air for twenty-four hours. Is it deliquescent? Is it hygroscopic? Test the liquid left in the generator with pink litmus. The reaction in the generator is thus expressed:

$$CaC_2 + H_2O = C_2H_2 + CaO.$$
$$CaO + H_2O = CaO_2H_2 \text{ (calcium hydroxide).}$$

5e. *Flame*

Flame is simply gas, burning freely. A non-volatile 284
combustible like carbon burns without flame. To observe some of the characteristics of flame, use the Bunsen burner, turned low, and the air shut off. Note the three different portions of the flame: First, the outer cone, pale blue, visible only at the base of the cone, where the light is feeble; second, the bright luminous cone; third, the dark interior cone. In the *outer cone* the combustion is complete, since there is free access of air. In the *middle luminous cone* the combustion is incomplete, since the air does not penetrate so far; but the temperature is high, and under these conditions the carbon-containing constituents of the gas undergo chemical change somewhat as seen in the destructive distillation of wood. This results in the separation of more or less carbon as very small solid particles. These are raised to a high temperature, white heat (incandescence),

before they come to the outer cone, where they burn to carbon dioxide.

285 Hold a cold object, like a porcelain dish, in this flame, and some of the carbon is deposited thereon (as soot) before it can reach the air and burn.

286 The *inner dark cone* contains unburned combustible gas, and the temperature is much lower. Insert the open end of a narrow glass tube in this cone, and the unburned gas may be drawn off and ignited at the upper end of the tube. Quickly thrust the head of a match through the outer cones and into the inner one. It does not take fire while held there, since the temperature is comparatively low and there is no air; but the wood of the match quickly burns at the edge of the flame. A piece of wire thrust horizontally through the flame is seen to become luminous first where it cuts the outer cones, while the portion in the inner cone remains dark for a few seconds.

287 With a few trials, a card, or a sheet of stiff paper may be lowered directly down upon the flame and quickly removed without taking fire, but it will show a ring of scorching where it has cut the outer cones. Lower a piece of wire gauze down upon the flame, and the dark central portion and the luminous ring are plainly seen.

288 In this experiment with the gauze it is seen that, although the gas is free to pass through the gauze, the flame is only on the lower side of it. The gas above the gauze may be reignited with a match.

Bring the gauze squarely down upon the flame quite to the burner, and the flame may thus be completely extinguished. Or, thrust the gauze through the flame horizontally and close to the burner so that there is no flame underneath it, then raise it to the tip of the cone, and the flame is likewise extinguished.

289 These effects are due to the fact that the gauze, being a good conductor of heat, reduces the temperature of the gas below its point of ignition. A valuable application of the fact is seen in the safety lamp,

invented by Sir Humphry Davy, which miners carry in coal mines where there are combustible gases and where there is consequently danger of explosive ignition by the lamp. The lamps are inclosed by a chimney of wire gauze which permits air to pass in, but no flame to pass outward.

Effect of temperature on luminosity.—The fact that the lowering of temperature lowers also the luminosity of flame may be seen by bringing against the flame a porcelain dish, or, better, an iron plate. The bright portion almost disappears, and hardly more than the pale blue is seen. 290

The Bunsen flame.—By opening the valves at the base of the chimney, so that air enters and mingles with the gas before it burns, the character of the flame is greatly changed. The presence of air in the interior of the flame brings about more complete combustion, the solid particles of carbon do not separate, luminosity nearly disappears, and no soot is deposited on cold objects. The luminosity may be momentarily restored by blowing any fine dust into the flame, such as iron dust, charcoal, or sodium carbonate. 291

The reduction of luminosity is not dependent on complete or increased combustion, since a gas containing no oxygen, such as nitrogen, produces the effect, if mixed with the gas before burning. In this case dilution and consequent reduction of temperature probably bring about the result. 292

Recall in this connection the character of the flame made by the use of the mouth blowpipe. (See No. 229, Part II, and Appendix, 21.)

6. NITROGEN

N.—13.93

Preparation.—The air is mainly a mixture of nitrogen and oxygen, and the removal of the latter gives the most available method of preparing nitrogen. 308

On a crucible lid, resting on a flat cork, and floating on the water in the tank, place a small piece of phosphorus.

Bringing the cork over the bridge of the tank, ignite the phosphorus and invert a beaker over the latter, letting the beaker rest upon the bridge. The phosphorus burns with a white smoke, which is the pentoxide of phosphorus (P_2O_5), and this is slowly absorbed by the water. The water rises in the beaker, taking the place of the oxygen thus removed, and the gas left is nitrogen. This may be poured upward under water into another beaker or collecting bottle, and the operation repeated until sufficient gas is obtained.

309 **Properties.**—Describe the gas, testing in the usual way as to solubility in water, as to action on red and on blue
310 litmus paper, and as to combustion with the taper.

310/1 NOTE.—Phosphorus must be handled with the greatest care, as it ignites very easily, and burns made by it are very painful and difficult to heal. It should never be cut except while it is under water; the piece to be ignited should be gently touched with filter paper to take up the adhering water. After the generation of nitrogen, any phosphorus left adhering to the lid should be carefully scraped off with the spatula and completely burned, so that there shall be *positively no phosphorus left unconsumed.*

6a. *Nitrogen Monoxide or Nitrous Oxide, N_2O*

312 **Preparation.**—Use the flask fitted with a stopper, and with a rubber and glass delivery tube. Place in this a charge of solid ammonium nitrate, NH_4NO_3, considerably more than enough to cover the bottom of the flask. Support the latter on the asbestos board and iron stand. It is well to pass the neck of the flask through the small ring to prevent its falling over. Apply heat, slowly at first, until a layer of melted salt covers the bottom of the flask. As the heating continues the salt apparently boils; this is really decomposition, and, when it is well started, the flame should be removed lest the action become too violent. Collect the gas over water. Remove the delivery tube from the water before the current of gas ceases. The gas is
313 nitrogen monoxide. Describe it, testing in the usual man-

ner as to solubility, action on litmus paper, and relation to 314
combustion.

EXPLANATORY NOTE.—Ammonium nitrate is decomposed by heat into nitrogen monoxide and water, and the reaction liberates heat. Write the equation.

6b. *Nitrogen Dioxide or Nitric Oxide, NO*

Preparation.—As in Exp. 47/b. 317

Examine **as to solubility.** After shaking in a test-tube 318
with water, slip a crystal of ferrous sulphate into the test-tube, opening it under water, and shake again.

As to action on litmus.—This can be observed only by 318/1
slipping the litmus paper under water into the tube or bottle containing a sample of the gas, since contact with air would entirely change its character.

As to combustion.—The usual test with the taper can 318/2
only be applied in opening the bottle to the air, hence the observed property really belongs to nitrogen tetroxide or to a mixture of the two oxides.

Make the combustion test likewise with a piece of ignited magnesium ribbon.

Save a portion of this gas for the next experiment.

6c. *Nitrogen Trioxide or Nitrous Anhydride, N_2O_3*

No experiments. 319 to 321

6d. *Nitrogen Tetroxide or Peroxide, N_2O_4 or NO_2*

Compare Experiments $47/_1$ and $47/_2$.

Pass some air into the bottle containing a sample of nitric oxide. Note the color, solubility, and action on blue 322 to
litmus paper of the tetroxide thus produced. 324

6e. *Nitrogen Pentoxide or Nitric Anhydride, N_2O_5*

No experiments. 325

6f. *Nitric Acid, HNO_3*

329 **Preparation.**—It is made by the combination of nitrogen pentoxide and water which takes place on contact; also by adding sulphuric acid to a nitrate—for example, potassium nitrate—and distilling the nitric acid thus liberated from the salt. (See Appendix, 18 B.) What is the other product in this reaction?

331 **Properties.**—Recall the reactions between nitric acid and ferrous sulphate (Exp. 317), and zinc (Exp. 41/2) and tin (Exp. 144).

Neutralize about one half test-tubeful of nitric acid from the side table (this is really a solution of nitric acid in water, about 63 per cent) with ammonium hydroxide, concentrate, and crystallize the salt.

331/1 Take a few of these crystals, or, without waiting for these, take some of the substance from the side table, and, heating on the spatula blade, touch a glowing coal to the melted salt.

In similar manner use a few crystals of sodium or potassium nitrate.

331/2 **Nitrohydrochloric acid.**—Cut off a small piece of your platinum wire. Boil this in a test-tube with a few drops of concentrated hydrochloric acid. Does it dissolve? Pour off the acid and try if the platinum dissolves in concentrated nitric acid. Then add to the latter a few drops of hydrochloric acid, and again boil. What evidence of reaction do you see? Does the platinum dissolve?

331/3 Explanatory Note.—In this reaction the hydrogen of the hydrochloric acid is oxidized to water by the nitric acid, chlorine is liberated, and the lower oxide of nitrogen produced. The reaction takes place only when the acids are concentrated, and the solvent action is due to the nascent chlorine. The mixture of the two acids is called nitrohydrochloric acid, or "aqua regia," as it was named in the early days of chemistry because of its powerful solvent action on gold and other metals.

6h. *Ammonia*, NH_3

Preparation.—Heat in a test-tube a piece of flannel or other woolen stuff. Note the odor of the gas and its action on litmus. 334

EXPLANATORY NOTE.—Wool, hair, and similar organic substances contain nitrogen, as does coal also; these, by dry distillation, yield ammonia or ammonialike substances. Ammonia, therefore, is a product in making illuminating gas by the distillation of coal.

Rub together in the mortar, or even in the palm of the hand, some sodium carbonate or some lime and an ammonium salt. What gas is liberated? Write the equation for the reaction. 334/1

In a flask fitted with stopper and delivery tube, heat some ammonium hydroxide solution. Collect the gas in a dry bottle; it is lighter than air. Is it soluble in water? What is its action on litmus? Does the taper or match burn in the gas? Does the gas itself burn? Test this by holding the tube which delivers the ammonia close to the flame, or by passing the gas into the openings at the base of the chimney. What do you infer are the products when ammonia burns? 334/2 335

7. OXYGEN

O.—15.88

Preparation.—Heat in a test-tube a small quantity of potassium chlorate, $KClO_3$ (about as much as could be heaped on a copper cent). Note the phenomenon, and test the gas at the mouth of the test-tube with a lighted splinter of wood, having care that nothing drops into the test-tube. In a similar manner heat about the same quantity of manganese dioxide. Are you able to get evidence of oxygen at the mouth of the tube? Now mix a little of the chlorate and dioxide, and heat as before and test the gas. Is the gas obtained more readily with or without the dioxide? 352

To obtain the gas in larger quantity for examination (if the reservoir supply is not at hand), prepare it as in Exp. 71 A, and collect in the smaller bottles. Recall the results of Exps. 71 A and 71/2.

353 to 357 Examine as to solubility, action on litmus, and relation to combustion, using a candle or splinter of wood; and if further illustration is desired, use a bit of sulphur ignited in the metal spoon.

NOTE.—After oxygen is evolved from the chlorate, $KClO_3$, potassium chloride is left. If the temperature is high enough, oxygen is obtained from the manganese dioxide alone, as expressed in the equation

$$3MnO_2 = Mn_3O_4 + 2O.$$

8. FLUORINE

F.—18.91

402 The preparation of this element is difficult, and it is practically impossible as a laboratory experiment.

Its compound with calcium, CaF_2, is a common mineral, 405 known as *calcium fluoride* or *fluor spar*. This is a salt of hydrofluoric acid, HF. By acting upon it with strong sulphuric acid, the former acid is liberated as a gas.

$$CaF_2 + H_2SO_4 = CaSO_4 + 2HF.$$

406 The hydrofluoric acid attacks and dissolves glass, and is used practically for etching. This may be illustrated as follows: Clean and dry a piece of window glass (use one of your glass covers); spread over it a thin film of melted paraffin, using the hot spatula blade to melt the latter. With the point of a pin or penknife expose the glass surface by tracing any chosen design through the paraffin. In a shallow lead dish place some powdered fluor spar, and mix this with sulphuric acid to a thin paste. A splinter of wood may serve as stirrer. Cover the dish with the glass plate, the protected side down, and leave for twenty-four hours.

Clean off the paraffin with hot water, and the design will be found etched into the glass. (Scrape the content of the lead dish into the waste jar.)

9. SODIUM

Na.—22.88

The element is supplied already prepared. Describe its 410
appearance. It decomposes water at ordinary temperature. To show this, wet a filter paper, spread it flat on the table, and lay on it a piece of red litmus paper. Place on the filter a piece of sodium, perhaps twice the size of a pin-head. (Have care not to touch sodium with wet fingers.) Describe the phenomenon. If the sodium does not take fire, let a drop of water fall upon it from a glass rod or the end of the finger. Note the action on the litmus. Explain the whole reaction. (Toward the end of the reaction the little globule of melted metal is likely to snap off the paper. Be careful that it is not thrown in the face, or elsewhere, to do harm.)

9c. *Sodium Hydroxide or Caustic Soda*

Recall what is already known of this substance from 420
previous experiments. Describe the solid hydroxide. Make a solution of it, a stick 4 or 5 centimeters long (1½ or 2 inches), in a beaker one half to three quarters filled with water. Observe what takes place during solution. Neutralize about three quarters of the solution thus obtained with hydrochloric acid, filter, concentrate, and crystallize. What is the appearance of the crystals? What is the substance? Write the equation for the neutralization.

With another portion of the sodium hydroxide solution, 420/1
observe and describe what takes place on adding a few drops of it to dilute solutions of these several salts in different test-tubes: Ammonium nitrate or chloride (heat, and note

odor), iron sulphate, copper sulphate, alum (i. e., aluminium sulphate), sodium nitrate, and perhaps other salts if at hand.

420/2 EXPLANATORY NOTE.—Sodium hydroxide is a base, and it is at least conceivable that, when added to a salt, it might combine with the acid of the latter, forming a new salt and leaving the base of the original salt wholly or partly deprived of its acid—i. e., leaving it in the condition of a hydroxide. Now, if this took place, and if either the new salt or the new hydroxide were insoluble in water, evidence of the change would be seen in precipitation. On the other hand, if the new substances were soluble, no precipitate would appear, and it might be difficult to get evidence as to the change. The fact is that the sodium salts are soluble, but the hydroxides of iron, copper, and aluminium are insoluble, and are the precipitates which are seen in these reactions. Ammonium hydroxide is soluble, hence no precipitate is seen with the ammonium salts; but if the mixture is boiled, ammonia is liberated, showing the presence of the hydroxide in the solution. With sodium nitrate, of course, there is no reaction. The reaction with iron sulphate is expressed in saying sodium hydroxide and iron sulphate become iron hydroxide and sodium sulphate. With this information let the student complete the second, third, and fourth of the following equations, in which the precipitates are underscored:

$$2NaOH + FeSO_4 = \underline{FeO_2H_2} + Na_2SO_4$$
$$NaOH + CuSO_4 = \underline{CuO_2H_2} +$$
$$NaOH + Al_2(SO_4)_3 = \underline{Al_2O_6H_6} +$$
$$NaOH + NH_4NO_3 = \underline{NH_4OH} +$$
$$NaOH + NaNO_3 = \text{no reaction.}$$

10. MAGNESIUM

Mg.—24.10

445 The element itself is supplied. Describe its appearance and recall whatever has been learned of it in previous experiments. By observation answer the following questions: Does magnesium melt? Does it burn? What is
446 the appearance of the product of combustion, MgO? Does the oxide dissolve in water? In hydrochloric acid?

Using strips of the ribbon, one or two centimeters long (one half to three quarters of an inch), ascertain if magnesium decomposes cold water or boiling water. (Have a

piece of red litmus paper in the water.) Does the hydroxide act as base or acid? Does the metal dissolve in very dilute hydrochloric acid? In very dilute nitric acid? In very dilute sulphuric acid?

Make a dilute solution of magnesium sulphate ($MgSO_4$), by dissolving the dry salt (from the side table), a lump about the size of a pea, in a test-tube nearly filled with water. Add to a small portion of this solution a few drops of sodium hydroxide solution. What is the precipitate? (See No. 420/2.) In a similar manner add to different portions of the magnesium sulphate solution a few drops of solutions of these several substances: ammonium hydroxide, ammonium chloride, ammonium carbonate, sodium carbonate, sodium phosphate. Magnesium hydroxide, carbonate, and phosphate are insoluble. With this information complete the following equations: 446/1

$$Mg + O =$$
$$Mg + H_2O = \underline{MgO_2H_2} +$$
$$Mg + HCl = \underline{MgCl_2} +$$
$$Mg + H_2SO_4 = MgSO_4 +$$
$$MgSO_4 + NaOH =$$
$$MgSO_4 + Na_2CO_3 = \underline{MgCO_3} +$$
$$MgSO_4 + Na_2HPO_4 = \underline{MgHPO_4} +$$

What is the valence of magnesium?

11. ALUMINIUM

Al.—26.9

The element itself is supplied. Describe its appearance. By trial learn if it melts, and if it burns. Does it decompose water, cold or boiling? Does it dissolve in dilute hydrochloric acid? In dilute nitric? In dilute sulphuric? In dilute solution of sodium hydroxide (boil)? What is the behavior of aluminium sulphate (alum) in dilute solution toward these several substances also in dilute solution: 451 to 454

sodium hydroxide (first in small proportion then in excess), ammonium hydroxide, ammonium chloride, sodium carbonate, sodium phosphate? Aluminium hydroxide and phosphate are insoluble in water; the chloride is soluble. The hydroxide dissolves in excess of sodium hydroxide, forming a soluble compound in which the former acts as acid, and the latter as base.

12. SILICON

Si.—28.2

EXPLANATORY NOTE.—It is not practicable to supply the element—not, indeed, even to show it. The dioxide, SiO_2, is, however, abundant
473 as common sand. This oxide acts as an acid-former, and when brought in contact with fused sodium carbonate it liberates carbon dioxide and combines with the base, forming a silicate, the composition of which may be sufficiently indicated by the formula Na_2OSiO_2 or Na_2SiO_3. This is soluble in water, and when hydrochloric acid is added to the solution the silicic acid, H_2OSiO_2, in turn is liberated and, being insoluble in water, is precipitated. The precipitate is, however, soluble in the hydrochloric acid, probably without chemical change, and may be reprecipitated by neutralizing the acid, HCl, with ammonium hydroxide.

474 Place a few grains of sand (powdered glass may be used in the same way) in a shallow hole of a piece of charcoal, cover the sand with five or six times its bulk of sodium carbonate, fuse thoroughly, using the blowpipe. Note the minute bubbles. What causes them? After sufficient fusion, remove the mixture from the charcoal, treat with hot water, filter, acidulate with hydrochloric acid, using litmus paper, and boil. If the precipitate does not appear, add ammonium hydroxide to slight alkalinity, and boil again. Describe the appearance of the precipitated silicic acid, H_2SiO_3. Filter the precipitate and dry it.

Complete the equations:

$$SiO_2 + Na_2CO_3 = Na_2SiO_3 +$$
$$Na_2SiO_3 + HCl = \underline{H_2SiO_3} +$$
$$H_2SiO_3(\text{heated}) = \underline{SiO_2} +$$

13. PHOSPHORUS

P.—30.8

Recall the use already made of phosphorus. It must be handled with the utmost caution, because of the danger of ignition. Burns produced by it are painful and difficult to heal.

Describe the appearance of yellow phosphorus. **486**

Place the porcelain lid and cork on a dry glass plate. **487** On the lid place a small fragment of phosphorus. Ignite the latter and cover it with a dry beaker. The oxide formed is mainly P_2O_5. Describe the burning and the product. Let the white oxide settle upon the glass plate. Observe the marked deliquescence. Touch blue litmus paper to the oxide. Is it an acid- or a base-forming oxide?

Phosphorus a reducing agent.—Into a test-tube about one **487/1** third filled with a dilute solution of silver nitrate, $AgNO_3$, drop a small piece of yellow phosphorus, about twice the size of a pinhead. Let stand for twenty-four hours. Minute crystals of metallic silver will be seen at the bottom of the test-tube. The liquid may be drained off and the crystals fused on the charcoal or asbestos to a small globule of silver.

Describe the appearance of the red phosphorus, and **490** compare it with the yellow as to inflammability.

EXPLANATORY NOTE.—Phosphorus pentoxide combines with water **494** to in three different ratios, forming three phosphoric acids, thus: **496**

$$P_2O_5 + 3H_2O = 2H_3PO_4 = \text{orthophosphoric acid.}$$
$$P_2O_5 + 2H_2O = H_4P_2O_7 = \text{pyrophosphoric acid.}$$
$$P_2O_5 + H_2O = 2HPO_3 = \text{metaphosphoric acid.}$$

When sodium orthophosphate, Na_2HPO_4, is sufficiently heated, it becomes the pyrophosphate, $Na_4P_2O_7$. Both are soluble in water, but the latter does not immediately pass back to the orthophosphate when in contact with water.

Taking a fragment of crystallized sodium orthophosphate, Na_2HPO_4, fuse it on charcoal, keeping it fused for some minutes. Remove the salt when cool, dissolve in a

little hot water, filter, and to the solution add a few drops of silver nitrate solution. Describe the precipitate, $Ag_4P_2O_7$. Dissolve a small piece of the sodium orthophosphate in water, and add to this a few drops of silver nitrate. Describe this precipitate, Ag_3PO_4. How has the fusion changed the orthophosphate? Write the equation for the reaction of fusion, also for the precipitations.

14. SULPHUR

S.—31.83

509 **Sulphur** was studied in considerable detail at the very first of the course, and it would seem unnecessary to repeat the experiments here. They should at least be recalled, and it may be well to write out a description from the earlier observations. These items may serve as outline: specific gravity, as to solubility in water, color, hardness, crystalline form from fusion, from solution in carbon disulphide, plastic allotropic form, fusion, combustion, behavior with iron, with zinc, and with lime.

510 and 511 **Hydrogen sulphide,** H_2S, also was studied somewhat, and these items should be recalled: formation by the action of hydrochloric acid on metallic sulphides, odor of gas, action on paper wet with lead acetate, behavior with strong nitric acid.

512 **The sulphides.**—Hydrogen sulphide is an acid, and in combining with ammonium hydroxide it forms the acid sulphide, NH_4HS, or the normal $(NH_4)_2S$. Persulphides also are formed, such as $(NH_4)_2S_2$ and $(NH_4)_2S_3$, the coefficient of S being 2 or more. The common reagent, yellow ammonium sulphide, much used in the laboratory, is made by saturating ammonium hydroxide with the gas, H_2S. It therefore contains more or less persulphide. With this solution of ammonium sulphide make the following experiments and describe the results: Add a small portion to these several solutions: hydrochloric acid, sodium nitrate, magnesium sulphate, alum, iron sulphate, copper sulphate, lead acetate, and, if at hand, to solutions containing arsenic, antimony, and cadmium.

Complete the following equations:

$$(NH_4)_2S + HCl =$$
$$(NH_4)_2S_2 + HCl = \underline{S} +$$
$$(NH_4)_2S + NaNO_3 =$$
$$(NH_4)_2S + MgSO_4 =$$
$$(NH_4)_2S + Al_2(SO_4)_3 = \underline{Al_2O_6H_6} +$$
$$(NH_4)_2S + FeSO_4 = \underline{FeS} +$$
$$(NH_4)_2S + CuSO_4 = \underline{CuS} +$$

What is the valence of sulphur in hydrogen sulphide?

Sulphur dioxide.—This substance is formed by burning 515
sulphur, but more conveniently by the action of hydrochloric acid on sodium sulphite, Na_2SO_3. Place some of the latter in the generator, add the acid gradually, and collect the gas by dry displacement, since it is soluble in
water and heavier than air. Test the gas for odor (it is 516
very irritating), solubility, action on the burning candle or match, weight as compared with air (as in the experiment with carbon dioxide No. 263), and action on litmus paper, both brief and somewhat prolonged.

To a portion of sodium sulphite solution add a few 517
drops of potassium permanganate, $KMnO_4$, and to another portion add a crystal of iodine. Describe what takes place.

Sulphur dioxide, sulphurous acid, H_2SO_3, and the sulphites, all tend more or less readily to take up oxygen and become the trioxide, sulphuric acid, H_2SO_4, and the sulphates respectively; the former therefore act as reducing agents.

Complete these equations: 517/1

$$S + O =$$
$$SO_2 + H_2O =$$
$$H_2SO_3 + NaOH = Na_2SO_3 +$$
$$Na_2SO_3 + HCl =$$
$$SO_2 + O =$$
$$H_2SO_3 + O =$$
$$Na_2SO_3 + O =$$
$$Na_2SO_3 + H_2O + I = 2HI +$$
$$Na_2SO_3 + K_2O.Mn_2O_7 + H_2O = 2KOH + 2MnO +$$

519 **Sulphuric acid.**—With sulphur trioxide it is not practicable to work, but its compound with water, *sulphuric acid*, $H_2O.SO_3$, or H_2SO_4, is a common reagent. Describe its appearance.

Taking about one half of a test-tubeful of the concentrated sulphuric acid, dilute it with about three times its volume of water. Note what takes place. CAUTION: *Make it a rule in diluting strong sulphuric acid always to add the acid to the water slowly and with constant stirring, and never the water to the acid.* Why?

521 Taking three equal portions of the dilute acid, neutralize one portion with a solution of sodium hydroxide, filter, concentrate, and crystallize. Neutralize the second portion in the same way, and to it add the third portion; filter, concentrate, and crystallize. Compare the crystallizations; dry a sample of each product on filter paper, and test it with litmus paper, red and blue. Let a sample of each lie exposed to the air for twenty-four hours.

519/1 To show the tendency of strong sulphuric acid to remove the constituents of water: having a small quantity of the acid in a test-tube, drop into it a splinter of pine wood, or a piece of filter paper. What takes place after some minutes?

521/1 Sulphate changed to sulphide: taking a crystal of sodium sulphate from the side table or from the product of the preceding experiment, No. 521, fuse it on the charcoal, first adding some sodium carbonate (use the reducing flame of the blowpipe). Remove the product from the charcoal, lay it upon a piece of filter paper wet with lead acetate, and let a drop of water fall upon it. What does the black stain indicate? A silver coin may be substituted for the lead paper.

521/2 **Solubility of sulphates.**—Add a few drops of sodium sulphate or of dilute sulphuric acid to a solution of lead acetate; also, if at hand, to the solution of a barium salt (e. g., $BaCl_2$), and of a strontium salt (e. g., $SrCl_2$).

Complete these equations:

$$SO_3 + H_2O =$$
$$H_2SO_4 + NaOH = Na_2SO_4 +$$
$$H_2SO_4 + NaOH = NaHSO_4 +$$
$$Na_2SO_4 + C = Na_2S + CO_2$$
$$H_2SO_4 + BaCl_2 = \underline{BaSO_4} +$$
$$Na_2SO_4 + SrCl_2 = \underline{SrSO_4} +$$
$$Na_2SO_4 + Pb(A)_2 = \underline{PbSO_4} +$$

15. CHLORINE

Cl.—35.18

This element is extremely disagreeable to deal with in large quantity. It is best that the illustrative experiments in the laboratory be only on the test-tube scale.

Preparation.—Hydrochloric acid is commonly the source 529
of chlorine. From this it is obtained by oxidizing the hydrogen to water, and thus liberating the chlorine. The methods differ only in the means used to oxidize.

(*a*) Place in a test-tube a small quantity of manganese 529/2
dioxide, MnO_2, about as much as could be heaped on a copper cent. Add to this about one quarter of a test-tubeful of concentrated hydrochloric acid and warm gently. Observe cautiously the odor, also the effect on wet litmus 530
paper of brief and then of somewhat prolonged exposure to the gas. Enough of the gas may be generated in the test-tube to show its color, and some of it may be poured into a second dry tube (it is heavier than air) in order to test as to its solubility in water.

Complete this equation by inserting the necessary coefficients:

$$MnO_2 + \quad HCl = MnCl_2 \text{ (sol. salt)} + \quad H_2O + \quad Cl.$$

(*b*) Place in a test-tube some potassium chlorate, $KClO_3$, 529/1
about the size of a pinhead, and add concentrated hydrochloric acid, a few drops. Note the evolution of gas, the

odor, color, etc. Dilute with water. Does the reaction continue? Complete the equation:

$$KClO_3 + \quad HCl = KCl \text{ (sol. salt)} + \quad H_2O + \quad Cl.$$

Hydrochloric Acid, HCl

534 **Preparation.**—Place some sodium chloride, NaCl, in a test-tube; a lump about the size of a pea will serve. Add to this a few drops of concentrated sulphuric acid, H_2SO_4, and warm. Note the odor of the gas, and its action on blue litmus paper. Hold at the mouth of the test-tube a glass rod wet with ammonium hydroxide. What causes the white fumes?

The properties of hydrochloric acid have been illustrated in its repeated use.

534/1 **Solubility of chlorides.**—Mix a few drops of hydrochloric acid, or of sodium chloride solution, with these several salts in dilute solution: lead acetate, $Pb(C_2H_3O_2)_2$; silver nitrate, $AgNO_3$; mercurous nitrate (if at hand), $HgNO_3$; magnesium sulphate, $MgSO_4$; iron sulphate, $FeSO_4$, etc. The first three of these bases form insoluble chlorides. The other chlorides are as a rule soluble.

Complete these equations:

$$NaCl + H_2SO_4 =$$
$$HCl + Pb(C_2H_3O_2)_2 =$$
$$HCl + AgNO_3 =$$
$$HCl + HgNO_3 =$$
$$HCl + MgSO_4 =$$

16. POTASSIUM

K.—38.82

552 and 553 If the metal potassium and its hydroxide, KOH, are at hand, experiments with them may be made exactly as with sodium and sodium hydroxide. But the results are so entirely similar that the observations may be omitted.

17. CALCIUM

Ca.—39.7

The element is not available for these experiments, 581
being difficult to obtain, but the oxide, lime, CaO, is readily procured. If quicklime is at hand, use it to observe the effect of contact with water. If only slaked lime, $Ca(OH)_2$, is provided, it will serve, except for this single observation. Learn by experiment if calcium hydroxide dissolves 582
in water. Does it act as base or as acid? What reaction takes place with carbon dioxide? Prepare a solution of calcium chloride, $CaCl_2$, by neutralizing with lime about one third of a test-tubeful of the acid, and filtering. With 584
this solution, observe the reactions with dilute solutions of these several substances: ammonium hydroxide, chloride, carbonate, and oxalate $(NH_4)_2C_2O_4$; sodium phosphate, and sodium or potassium sulphate or dilute sulphuric acid. Complete the following equations:

$$CaO + H_2O =$$
$$Ca(OH)_2 + CO_2 =$$
$$Ca(OH)_2 + HCl =$$
$$CaCl_2 + (NH_4)_2CO_3 =$$
$$CaCl_2 + (NH_4)_2C_2O_4 =$$
$$CaCl_2 + Na_2HPO_4 =$$
$$CaCl_2 + Na_2SO_4 =$$

23. IRON

Fe.—55.6

Several experiments with iron and its salts have already 596
been performed, and should be recalled here. With a sample of iron, learn by trial if it melts or burns in the flame of the Bunsen burner or in that of the blowpipe. Does it decompose water, cold or boiling? Does it dissolve in 598
dilute hydrochloric acid (recall other experiments)? In dilute nitric acid? In dilute sulphuric acid?

Take about a test-tubeful of hydrochloric acid, dissolve in this as much iron as it will take up, keeping some excess of iron present (a nail serves well). Filter, concen-
600 trate, and crystallize the ferrous chloride.

As alternative with the preceding, dilute sulphuric acid may be used and the ferrous sulphate may be crystallized.

To a dilute solution of ferrous salt freshly made, add ammonium hydroxide solution. What is the precipitate?

600/1 To a dilute solution of ferrous salt add a few drops of concentrated nitric acid, boil a few seconds, then add ammonium hydroxide to alkaline reaction. What is the pre-
601 cipitate? [Ferric hydroxide, $Fe_2(OH)_6$ or $Fe(OH)_3$, similar to iron rust.] What takes place when solution of ammonium sulphide is added to a ferrous solution? Recall the experiment made previously in studying the sulphides.

EXPLANATORY NOTE.—The ferrous salts when in solution oxidize readily to ferric salts even by the action of the air, more quickly by the action of nitric acid. The color changes in consequence, and the precipitates with ammonium hydroxide and other reagents are different.

Complete the following equations:

$$Fe + HCl = FeCl_2 +$$
$$Fe + H_2SO_4 = FeSO_4 +$$
$$FeSO_4 + HNO_3 = Fe_2(SO_4)_3 + NO +$$
$$FeCl_2 + NH_4OH = Fe(OH)_2 +$$
$$FeCl_2 + NH_4HS = FeS +$$
$$FeCl_3 + NH_4HS = FeS +$$
$$FeCl_3 + NH_4OH = Fe(OH)_3 +$$

APPENDIX

Weighing.—Substances which corrode metal should be 1
weighed on glass, and general care should be taken to protect the metallic parts of the balance from injury. Things when weighed should not be hot, but should be at the temperature of the room. During the more exact weighing, care should be taken that currents of air do not interfere with the swinging of the beam. The equilibrium should be tested while the beam is in motion by the equality of swing to either side of the zero point, *S*, on the scale. Generally the equilibrium should be tried with nothing in the pans before the actual weighing. In using balances of the type A or B (Fig. 5 or Fig. 6), it is best to put the object to be weighed on the left-hand pan, and the weights, or counterpoise, on the right. The weights should be placed in the pan for trial in regular succession, until one is found which is too heavy and one which is too light, and finally the exact counterpoise is found. It is well to read the weights first from the vacant places in the box, record the total, then read from the weights themselves as they are taken from the pan, the largest first. Care must be taken to return each weight to its proper place, to guard against the loss of any, and to leave the balance clean and in proper condition for the next weighing.

The balance of the type A is for the heavier weighings, and will hardly respond to less than 0.2 or 0.3 of a gram. The balance B should indicate 0.01 of a gram. The equilibrium is tested by pressing down the lever, *L*, which raises

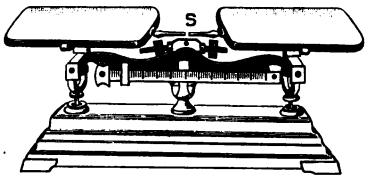

FIG. 5.—Balance A.

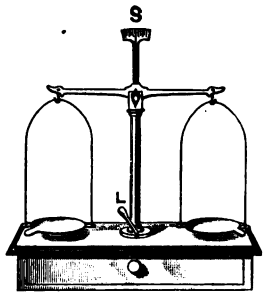

FIG. 6.—Balance B.

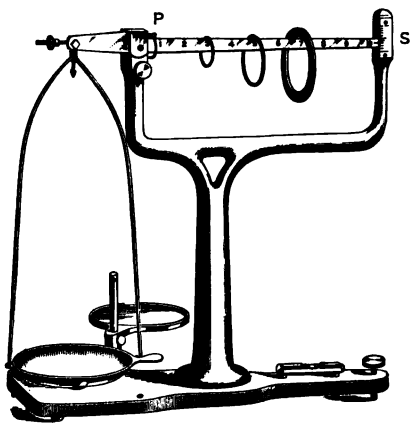

FIG. 7.—Balance C.

Weights.

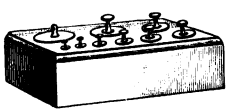

FIG. 8.

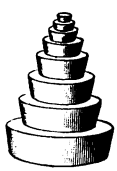

FIG. 9.

FIG. 10.

the beam so that it is free to swing. The balance C has but one pan, and the weights are in the form of rings which slide along the beam without removal therefrom, but when not in use hang upont he peg, *P*. The largest ring gives grams in tens, the next smaller in units, the third in tenths, and the fourth in hundredths. So the total reading shown in the figure is 75.30 grams.

Heating a test-tube, Fig. 11.—A strip of paper is folded 2
two or three times upon itself and wrapped around the test-tube to serve as holder, or, better, a strip of asbestos is thus used.

FIG. 11.

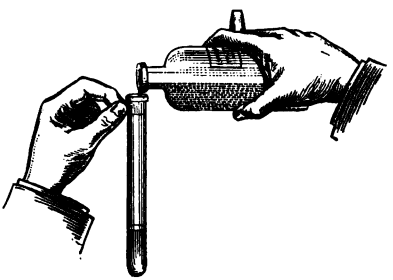

FIG. 12.

Pouring from a reagent bottle, Fig. 12.—The stopper is 3
taken between the fingers of the right or left hand as preferred, and withdrawn from the bottle. This is to avoid laying the stopper on the table.

Inverting and shaking a test-tube, Fig. 13. 4

The mortar and pestle, Fig. 14. 5

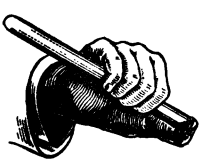

FIG. 13.

FIG. 14.

6 *The gas generator*, Fig. 15.—*T*, Thistle-tube; *D*, delivery tube. It is well to wet glass tubing when putting it through stoppers or connecting it with rubber hose.

7 *To cut glass tubing.*—Make a scratch with a three-edged file where it is desired the tube shall break. Hold the tube in both hands, the thumbs brought together on the opposite side from the scratch. Pull the hands apart in the direction of the tube's axis, with only a *very slight* bending motion. If the tube is short or the glass thin, it is well to hold it in one or two folds of the towel. Fig. 16.

8 *To smooth the sharp edge* of a cut tube, hold the end in the gas flame until the glass softens. Fig. 17.

9 *To bend* glass tubing, hold it in the flame, preferably a wide one, twirling it between the thumb and fingers until the glass is thoroughly softened, then slowly bend to the desired angle. The bend should be smoothly curved, as seen in the figure. If the heating is insufficient, or the bending too hasty, the glass wrinkles, and the tube is likely to break. It is well to pass the tube through the flame once or twice after it is bent, so that the cooling may be slow. Fig. 18.

10 *To draw out and close* glass tubing, hold it in the flame until the glass is well softened, then draw apart slowly and to the desired extent, and finally heat it still more at the separating point and draw again. Fig. 19.

11 *Heating a crucible*, Fig. 20.—The crucible, *C*, rests on the pipestem triangle, *T*, and this in turn on the iron ring of the stand, *S*. The tongs, *X*, are used to handle the crucible or its lid while hot.

12 *Evaporation to dryness in the porcelain dish*, *D*, Fig. 21.—When there is danger of spattering, it is well to hold the burner in the hand, and to touch the tip of the small flame for a moment to the bottom of the dish. The purpose is to heat, but not enough to cause the formation of bubbles of vapor. Constant stirring with the rod, *R*, is important.

FIG. 15.

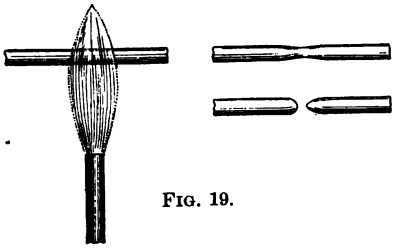

FIG. 19.

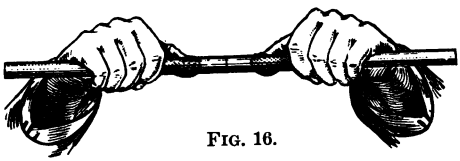

FIG. 16.

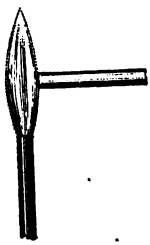

FIG. 17.

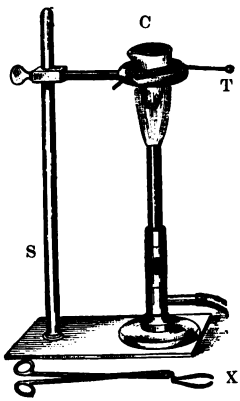

FIG. 20.

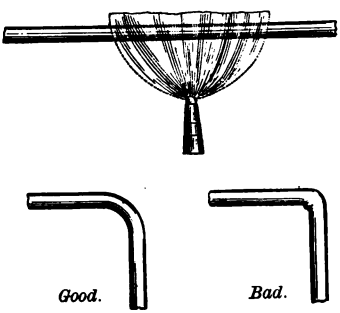

FIG. 18.

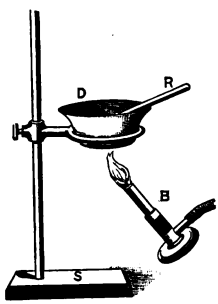

FIG. 21.

13 *Filtration.*—Prepare the filter paper by folding it across two diameters at right angles to each other, then separating the quadrants so as to form a cone, *d*. The cone should be fitted into the funnel and then wet, so that the paper adheres to the glass. Fig. 22.

Fig. 23.—Filtering, pouring the liquid from the beaker, *B*, using the rod, *R*, upon the funnel, *F*; showing the stem of the funnel lying against the wall of the beaker, so that the liquid which runs through may not cause spattering by dropping into the middle of the beaker.

14 *The graduated cylinder*, Fig. 24, to use in measuring liquids; content, fifty cubic centimeters (c. c.).

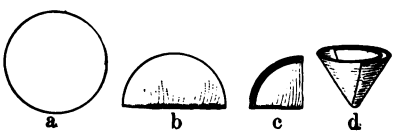

FIG. 22.

FIG. 23.

FIG. 24.

The thermometer.—The measurement of temperature is made with the centigrade thermometer. The fixed points of this are the boiling point of water and the melting point of ice. The latter is made 0° of the scale, and the former is made 100°, the space between the two points being divided into one hundred equal parts, called degrees. In the Fahrenheit thermometer, which is more commonly used in matters other than scientific, these two points are made 32° and 212° respectively. The relation between the two scales is made clear in the diagram. Fig. 25. 15A

Thermometers are fragile and expensive, and must be used with care.

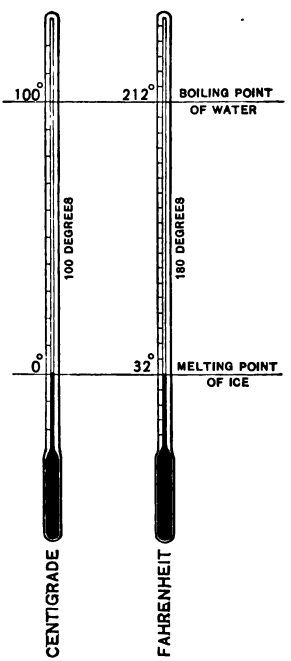

FIG. 25.

15B *The barometer*, Fig. 26.—The long tube is completely filled with mercury, then inverted in the shallow dish of mercury. The atmospheric pressure on the free surface of the mercury sustains a column of varying length, the normal being 760 millimeters. Fig. 27, the siphon form of barometer.

FIG. 26.

FIG. 27.

Melting-point determination.—Fig. 28, the sample tube, *ST*, attached to the thermometer by a rubber band. The sample tube is prepared by drawing out, as shown in Appendix No. 10. 16

Fig. 29.—The thermometer and tube suspended in a beaker of water; stirring rod, *R*.

FIG. 28.

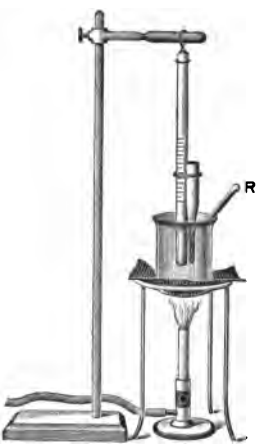

FIG. 29.

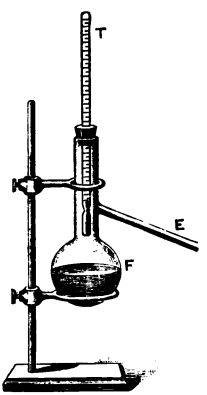

FIG. 30.

Boiling-point determination.—Fig. 30, the boiling flask, *F*, supported on the iron stand, the neck of the flask passed through the smaller ring; thermometer, *T*, passed through the stopper, the bulb being well above the liquid but below the exit tube, *E*. It is well always to wet the thermometer before passing it through the hole of the stopper; it thus slides more easily, with less danger of breaking. 17

18 *Distillation.*—Fig. 31, a common form of distilling apparatus for the laboratory. Flask, *F*; condenser, *C*; condensing water flowing in at *E*, and out at *O*; receiver, *R*; thermometer, *T*.

18B Fig. 32, another form of distilling apparatus, showing a retort, *R*.

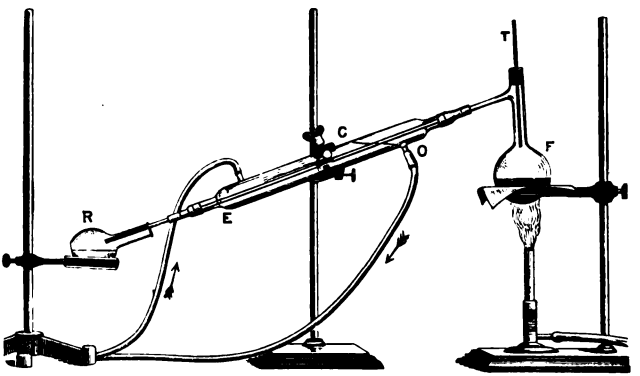

FIG. 31.

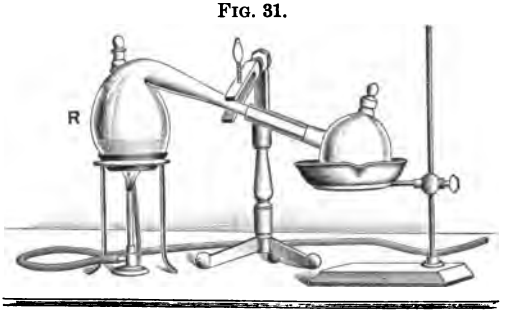

FIG. 32.

19 *Manipulation of gases.*—I. Fig. 33, the collection of gas over water. *D*, delivery tube; *B*, bridge on which the collecting vessels are supported; *T*, water-tank.

II. Fig. 34.—Pouring gas from one vessel to another. *B* is first filled with water, covered with a glass plate or piece of card board, and inverted in the tank. *A* contains the gas which is poured upward into the vessel *B*.

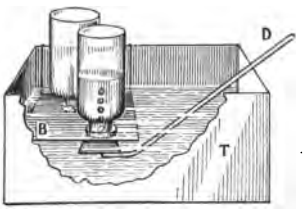

FIG. 33.

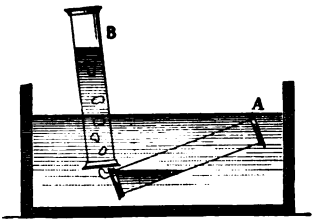

FIG. 34.

III. Fig. 35.—A bottle of gas removed from the tank for examination.

IV. Fig. 36.—Testing a gas with a lighted taper.

FIG. 35.

FIG. 36.

V. Fig. 37.—Collecting a gas heavier than air by dry displacement.

VI. Fig. 38.—Collecting a gas lighter than air by dry displacement.

FIG. 37.

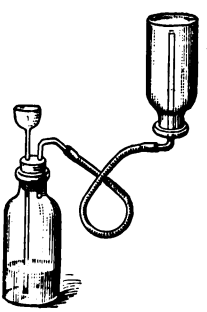

FIG. 38.

Chap II Law IV
Equivalent Proportions

754 mm. 273°
19 mm. 19°
735 mm. 292°

$$2421 \times \frac{735}{760} = 2340 \text{ c.c.}$$

$$2340 \times \frac{273}{292} = 2188 \text{ c.c.}$$

```
   2421
    735
  12105
   7263
  16947
760/1779435 (2340
    152
     259
     228
      314
      304
       1035
```

```
   2340
    273
   7020
  1638
  468
292/638820 (2188.
    584
     548
     292
     2562
     2336
      2260
```

```
  2188
  .0899
  19692
 19692
175104
196701 2
```

Dec 1st

Hydrogen & Magnesium.
1st Determination.

Temp. = 19°

Barometer = 754 mm.

Vap. Tens. of water = 19 mm. Wt of Mg = 2.40 gms.

Tot. Cap. Bottle 14 = 2554 c.c.

Water Residue = 101 gms.

Vol. gas collected = 2453

Vol. acid used = 32 c.c.

Vol. gas generated = 2421 c.c.

$2421 \times \frac{735}{760}$ = Vol. at 760 mm. = 2340 c.c.

$2340 \times \frac{273}{292}$ = Vol. at 760 mm. & 0° = 2188 c.c.

2.188 x .0899 = .197 gms. H from 2.40 gms. Mg

21 *The use of the mouth blowpipe.*—This, to be satisfactory, requires practice. It is necessary to blow continuously for some length of time, much longer than can be done in a single exhalation. But it is possible to use the cheeks like bellows in such a way that inhalation through the nostrils may go on without interrupting the current of air through the lips. Repeated trials, with some patience, will show better than words can how this is done.

I. To make the oxidizing flame: Insert the tip of the pipe in the gas flame (the yellow flame serves best), and blow a stream of air to the left hand and downward, so that you have a sharp, well defined tongue of blue flame. The bead should be held at the extreme tip of this, in order to have high temperature and free access of air. Fig. 39.

II. To make the reducing flame: Hold the tip of the pipe just at the edge of the flame and to the right, and blow more gently than before, so that you have a flame less contracted than the other and slightly yellow. The bead should be held well within the flame, near the tip of the inner cone, where there is high temperature, and where at the same time the bead is surrounded by hot, combustible gas with no access of air. Fig. 40.

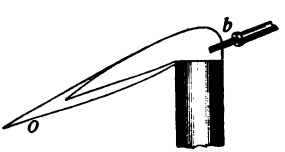

FIG. 39.

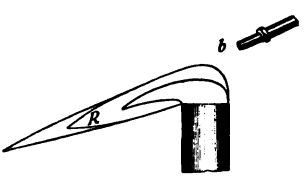

FIG. 40.

A Few Data of Frequent Use 22

One liter of air, at 0° and 760 millimeters, weighs 1.293 grams.

One liter of hydrogen, at 0° and 760 millimeters, weighs 0.0899 of a gram.

One liter of carbon dioxide, at 0° and 760 millimeters, weighs 1.977 grams.

Specific gravity of air (H = 1), 14.4.

One cubic centimeter of water, at 4°, weighs 1 gram.

One gram = $\frac{1}{30}$ of an ounce approximately; more closely = 0.035 of an ounce.

One gram = 15.4 grains.

One kilogram = $2\frac{1}{5}$ pounds approximately, more closely = 2.205 pounds.

One ounce (avoirdupois) = 28.35 grams.

One pound (avoirdupois) = 453.6 grams.

One liter = 1,000 cubic centimeters = 1.06 quarts (wine).

One quart (wine) = 0.95 of a liter.

One meter = 3.28 feet = 39.37 inches.

One millimeter = 0.00328 of a foot = 0.03937 of an inch.

One foot = 30.5 centimeters.

One inch = 25.4 millimeters.

Comparison of units, Fig. 41.

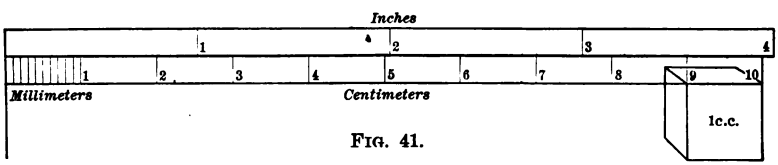

FIG. 41.

Pressure of water vapor in millimeters of mercury:

At 18° C. = 15 mm.	At 21° C. = 18 mm.
At 19° C. = 16 mm.	At 22° C. = 20 mm.
At 20° C. = 17 mm.	At 23° C. = 21 mm.

23 *A List of the More Common Acids (referred to in this Work), grouped according to Basicity*

Monobasic	HNO_3, Nitric.
	HCl, Hydrochloric.
	$HC_2H_3O_2$, Acetic.
Dibasic	H_2CO_3, Carbonic.
	H_2SiO_3, Silicic.
	H_2S, Hydrosulphuric.
	H_2SO_3, Sulphurous.
	H_2SO_4, Sulphuric.
	$H_2C_2O_4$, Oxalic.
Tribasic	H_3PO_4, Phosphoric.

24 *As to the Solubility of the Salts of these Acids*

The nitrates.—All soluble.

The chlorides.—The insoluble are silver, mercury (ous), and lead (slightly soluble).

The acetates.—Generally soluble.

The carbonates.—The alkali carbonates are soluble, others insoluble.

The silicates.—Insoluble, except the alkalies.

The sulphides.—Insoluble, except the alkalies and magnesium, calcium, strontium, and barium.

The sulphates.—Soluble, except calcium (slightly), strontium, barium, and lead.

The phosphates.—Generally insoluble, except the alkalies.

The hydroxides (metallic).—Generally insoluble, except the alkalies and calcium, strontium, and barium.

THE END

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